

# STIC Search Report

## STIC Database Tracking Number 195465

TO: Rei-Tsang Shiao Location: rem/5A10/5C18

Art Unit : 1626 July 24, 2006

Case Serial Number: 10/813822

From: Usha Shrestha Location: EIC 1700 REMSEN 4B28

Phone: 571/272-3519

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Search Notes	
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Other (specify)

## Scientific and Technical Information Center

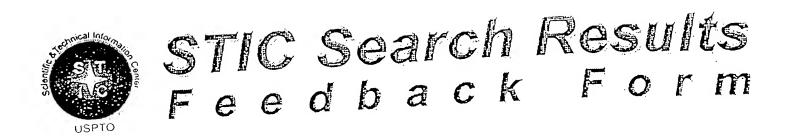
SEARCH REQUEST FORM Requester's Full Name: (Mailbox #): 5A10/ Results Format Preferred (circle): PAPER Location (Bldg/Room#): To ensure an efficient and quality search, please attach a copy of the cover sheet, claims, and abstract or fill out the following: Title of Invention: Inventors (please provide full names): Earliest Priority Date: Search Topic: Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched. Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc., if known. \*For Sequence Searches Only\* Please include all pertinent information (parent, child, divisional, or issued patent numbers) along with the I such a proon of whing apol of ford (I)
by: (see claim 9-16) cts (Cts)3 0 -0c-c-(H)n-0-N-cH-P-0c++5)2 - k C(Cts)3 0c21+5 K. X is \* Z is CHz=CHCHzz I Sul a prom of motor opd of claim 13, 14, 15 \* A is OR! Vendors and cost where applicable STAFF USE ONLY Type of Search STN NA Sequence (#) Searcher: Questel/Orbit \_\_ Lexis/Nexis AA Sequence (#) Searcher Phone #: Westlaw WWW/Internet Structure (#) In-house sequence systems Date Searcher Picked Up: Score/Length Commercial Oligomer Encode/Transl Interference Litigation . Date Completed:

Fulltext

Other

'Searcher Prep & Review Time: \_\_\_\_

Online Time:



# ACCEPANO)

Questions about the scope or the results of the search? Contact the EIC searcher or contact:

Kathleen Fuller, EIC 1700 Team Leader 571/272-2505 REMSEN 4B28

Voluntary Results Feedbails हिल्ला
<ul> <li>I am an examiner in Workgroup: Example: 1713</li> <li>Relevant prior art found, search results used as follows:</li> </ul>
102 rejection
103 rejection
Cited as being of interest.
Helped examiner better understand the invention.  Helped examiner better understand the invention.
Helped examiner better understand the state of the art in their technology.  Helped examiner better understand the state of the art in their technology.
Types of relevant prior art found
Foreign Patent(s)
[1] Non-Patent Literature (journal articles, conference proceedings, new product announcements etc.)
> Relevant prior art not found:
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<ul> <li>Results verified the tack of relevant prior are (1997).</li> <li>Results were not useful in determining patentability or understanding the invention.</li> </ul>

ARKEMA INC

(withdrawn) The alkoxyamine of Claim 1 wherein said alkoxyamine is 2-Methyl-2-[N-tert-butyl-N-7. (1-diethoxyphosphoryl-2,2-dimethylpropyl)aminoxy]propionic acid:

(withdrawn) The alkoxyamine of Claim 1 wherein said alkoxyamine is 2-Methyl-2-[N-tert-butyl-N-8. (1-diethoxyphosphoryl-2,2-dimethylpropyl)aminoxy]propionyl chloride:

(previously presented) A method for preparing a polymerised or non-polymerized mono- or polyalkoxyamine comprising reacting an alkoxyamine of formula (I):

in which A represents a hydroxyl radical, a radical R1O- in which R1 represents a linear or branched alkyl residue containing a number of carbon atoms ranging from 1 to 6; a radical MeO- in which Me represents an alkali metal; an H4N<sup>+</sup>-, Bu<sub>4</sub>N<sup>+</sup>- or Bu<sub>3</sub>HN<sup>+</sup>- radical; a chlorine atom; R represents a hydrogen atom or a methyl radical; M is a free-radical-polymerizable vinyl monomer sequence; n is an integer that may be equal to 0; to form a polymerised or nonpolymerized mono- or polyalkoxyamine of the formula (II):

$$Z = \begin{bmatrix} CH_{3} & C(CH_{3})_{3} & O \\ CC-C & (M)_{n} & CH-P-OC_{2}H_{5} \\ C(CH_{3})_{3} & OC_{2}H_{5} \end{bmatrix}_{X}$$

in which R and n have the same meaning as in formula (I); x is an integer at least equal to 1; Z represents a mono- or polyfunctional structure chosen from the structures given below in a non-limiting manner: CH<sub>2</sub>=CH-CH<sub>2</sub>-O-, CH<sub>2</sub>=CH-CH<sub>2</sub>-NH-, CH<sub>3</sub>-(OCH<sub>2</sub>CH<sub>2</sub>)<sub>p</sub>-O-, -O-(CH<sub>2</sub>)<sub>q</sub>-O-, p and q being integers at least equal to one, or more generally derived from compounds such as alcohols, polyols, amines, polyamines, epoxides, polyepoxides, esters, polyesters, amides, polyamides, imines, polyimines, polycarbonates, polyurethanes and silicones.

- 10. (previously presented) The method of Claim 9 wherein said alkali metal ME is selected from the group consisting of Li, Na, K, and mixtures thereof.
- 11. (previously presented) The method of claim 9 in which M is styrene, substituted styrenes, dienes, acrylic monomers, methacrylic monomers, acrylonitrile, acrylamide and its derivatives, vinylpyrrolidinone or a mixture of at least two abovementioned monomers.
- 12. (previously presented) The method of Claim 11 wherein the acrylic monomer is selected from the group consisting of acrylic acid or alkyl acrylates and mixtures thereof; and the methacrylic monomer is selected from the group consisting of methacrylic acid or alkyl methacrylates and mixtures thereof.
- (previously presented) The method of Claim 9 wherein said method forms allyl 2-[N-tert-butyl-N-(1-diethoxyphosphoryl-2,2-dimethylpropyl)aminoxy]propionate:

14. (previously presented) The method of Claim 9 wherein said method forms N-allyl-2-[N-tert-butyl-N-(1-diethoxyphosphoryl-2,2-dimethylpropyl)aminoxy]propionamide:

(previously presented) The method of Claim 9 wherein said method forms a dialkoxyamine of

16. (previously presented) The method of claim 9 wherein said method forms a compound of formula (II) in which x=1, n=0, R=CH<sub>3</sub> and Z=CH<sub>3</sub>(OCH<sub>2</sub>CH<sub>2</sub>)<sub>p</sub>O-.

g of the acid form of the alkoxyamine referred to as AA-SG1 in the form of a white powder (yield = 90%).

<u>Characterization</u> of 2-[N-tert-butyl-N-(1-diethoxyphosphoryl-2,2-dimethylpropyl)-aminoxy]propionic acid:

 $m.p. = 145^{\circ}C$ 

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<sup>31</sup>P NMR (121.59 MHz, CDCl<sub>3</sub>): □ 27.65 (s, Dia I, 65%). 24.60 (s, Dia II, 35%).

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): Dia I.  $\Box$  4.68 (q, J = 6 Hz, 1H), 3.90-4.35 (m, 4H), 3.38 (d, J = 27 Hz, 1H), 1.61 (d, J = 6 Hz, 3H), 1.34 (m, 6H), 1.20 (s, 9H), 1.19 (s, 9H). Dia II.  $\Box$  4.54 (q, J = 9 Hz, 1H), 3.90-4.35 (m, 4H), 3.38 (d, J = 27 Hz, 1H), 1.49 (d, J = 9 Hz, 3H), 1.31 (t, J = 9Hz, 6H), 1.17 (s, 9H), 1.12 (s, 9H).

<sup>13</sup>C NMR (75.54 MHz, CDCl<sub>3</sub>): Dia I. □ 174.17 (s, COOH), 81.46 (s, CH-O), 68.12 (d, J = 139 Hz, CH-P), 62.53 (s, N-C(CH<sub>3</sub>)<sub>3</sub>), 62.65 (d, J = 5.28 Hz, CH<sub>2</sub>), 59.86 (d, J = 7.55 Hz, CH<sub>2</sub>), 35.54 (d, J = 4.53 Hz, CH-C(CH<sub>3</sub>)<sub>3</sub>), 30.24 (d, J = 6.8 Hz, CH-C(CH<sub>3</sub>)<sub>3</sub>), 27.80 (s, N-C(CH<sub>3</sub>)<sub>3</sub>), 19.35 (s, CH-CH<sub>3</sub>), 16.31 (d, J = 5.29 Hz, CH<sub>2</sub>CH<sub>3</sub>), 16.04 (d, J = 6.8 Hz, CH<sub>2</sub>CH<sub>3</sub>). Dia II. □ 174.78 (s, COOH), 81.31 (s, CH-O), 69.47 (d, J = 141.26 Hz, CH-P), 62.53 (s, N-C(CH<sub>3</sub>)<sub>3</sub>), 62.22 (d, J = 6.8 Hz, CH<sub>2</sub>), 59.86 (d, J = 7.55 Hz, CH<sub>2</sub>), 35.59 (d, J = 2.26 Hz, CH-C(CH<sub>3</sub>)<sub>3</sub>), 29.85 (d, J = 6.04 Hz, CH-C(CH<sub>3</sub>)<sub>3</sub>), 27.72 (s, N-C(CH<sub>3</sub>)<sub>3</sub>), 18.43 (s, CH-CH<sub>3</sub>), 16.35 (d, J = 6.8 Hz, CH<sub>2</sub>CH<sub>3</sub>), 16.13 (d, J = 6.8 Hz, CH<sub>2</sub>CH<sub>3</sub>).

## 25 **Example 2**:

Esterification of AA-SG1

30 2 g of AA-SG1 (5.4 mmol) dissolved in 25 ml of dichloromethane predried over molecular sieves are placed in a 100 ml round-bottomed flask under a nitrogen atmosphere. 1.9 g of thionyl chloride (16.2 mmol) are added and the mixture is left to react for 45 minutes at room temperature. The reaction mixture is evaporated under vacuum to give the acid chloride of the alkoxyamine in the form of an oil, which is used in the subsequent synthesis without further purification.

The acid chloride obtained above is redissolved in 30 ml of ethyl ether (predried by distillation over sodium-benzophenone). A mixture containing 0.62 g of allyl alcohol (10.8 mmol), 0.55 g of triethylamine (5.4 mmol), 0.13 g of 4-dimethylaminopyridine (1.1 mmol) and 10 ml of ether is added thereto at room temperature. The mixture is left to react for 2 hours at room temperature. The reaction mixture is filtered, washed with aqueous 0.1 M HCl solution and then washed with aqueous 5% potassium bicarbonate solution. The organic phase is evaporated to give 1.53 g of the allylic amide of the alkoxyamine AA-SG1 (yield = 60%).

Characterization of allyl 2-[N-tert-butyl-N-(1-diethoxyphosphoryl-2,2-dimethylpropyl)aminoxylpropionate:

<sup>31</sup>P NMR (121.59 MHz, CDCl<sub>3</sub>): ☐ 23.23 (s, Dia I, 80%). 22.61 (s, Dia II, 20%).

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): □ 5.96-5.87 (m, 2H, dia I+II), 5.37-5.23 (m, 4H, dia I+II), 4.64-4.58 (m, 6H, dia I+II), 4.25-3.93 (m, 8H, dia I+II), 3.37 (d, J = 27 Hz, 1H, dia II), 3.27 (d, J = 24 Hz, dia I), 1.53 (d, J = 9 Hz, 3H, dia I), 1.50 (d, J = 6 Hz, 3H, dia II), 1.36-1.27 (m, 12H, dia I+II), 1.17 (s, 9H, dia II), 1.16 (s, 9H, dia I), 1.14 (s, 9H, dia II), 1.11 (s, 9H, dia I).

25 <sup>13</sup>C NMR (75.54 MHz, CDCl3): Dia I. □ 173.43 (s, CO), 131.69 (s, CH=CH<sub>2</sub>), 118.50 (s, CH=CH<sub>2</sub>), 82.49 (s, CH-ON), 69.51 (d, J = 139.75 Hz, CH-P), 64.90 (s, O-CH<sub>2</sub>-CH), 61.71 (d, J = 6.04 Hz, CH<sub>2</sub>), 61.52 (s, N-C(CH<sub>3</sub>)<sub>3</sub>), 58.67 (d, J = 7.55 Hz, CH<sub>2</sub>), 35.45 (d, J = 5.28 Hz, CH-C(CH<sub>3</sub>)<sub>3</sub>), 27.81 (s, N-C(CH<sub>3</sub>)<sub>3</sub>), 19.19 (s, CH-CH<sub>3</sub>), 16.40 (d, J = 5.29 Hz, CH<sub>2</sub>CH<sub>3</sub>), 16.10 (d, J = 6.8 Hz, CH<sub>2</sub>CH<sub>3</sub>). Dia II. □ 172.03 (s, CO), 132.06 (s, CH=CH<sub>2</sub>), 117.97 (s, CH=CH<sub>2</sub>), 82.49 (s, CH-ON).69.17 (d, J = 139.75 Hz, CH-P), 64.83 (s, O-CH<sub>2</sub>-CH), 61.81 (d, J = 8.3 Hz, CH<sub>2</sub>), 61.27 (s, N-C(CH<sub>3</sub>)<sub>3</sub>), 58.82 (d, J = 6.8 Hz, CH<sub>2</sub>), 35.10 (d, J = 5.28 Hz, CH-C(CH<sub>3</sub>)<sub>3</sub>), 30.17 (d, J = 6.04 Hz, CH-C(CH<sub>3</sub>)<sub>3</sub>), 27.87 (s, N-C(CH<sub>3</sub>)<sub>3</sub>), 17.73 (s, CH-CH<sub>3</sub>), 15.80 (d, J = 6.8 Hz, CH<sub>2</sub>CH<sub>3</sub>), 15.77 (d, J = 6.8 Hz, CH<sub>2</sub>CH<sub>3</sub>).

/ Example 3:

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**Amidation of AA-SG1** 

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The acid chloride of the alkoxyamine AA-SG1 is synthesized in the same manner as in Example 2.

2.1 g of acid chloride (5.4 mmol) are dissolved in 30 ml of ethyl ether. A mixture containing 0.62 g of allylamine (10.8 mmol), 0.55g of triethylamine (5.4 mmol), 0.13 g of 4-dimethylaminopyridine (1.1 mmol) and 10 ml of ether is added at room temperature. The mixture is left to react for two hours at room temperature. The reaction mixture is filtered, washed with aqueous 0.1 M HCl solution and then washed with aqueous 5% potassium bicarbonate solution. The organic phase is evaporated to give 1.53 g of the allylic amide of the alkoxyamine AA-SG1 (yield =70%)

<u>Characterization of N-allyl-2-[N-tert-butyl-N-(1-diethoxyphosphoryl-2,2-dimethyl-propyl)aminoxylpropionamide</u>:

<sup>31</sup>P NMR (121.59 MHz, CDCl<sub>3</sub>):  $\Box$  27.42 (s, Dia I, 35%). 27.05 (s, Dia II, 65%).

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): Dia I □ 8.61 (b, NH, 1H), 5.96-5.83 (m, 1H), 5.19 (dq,  $J_{HH}$  = 1.5 Hz,  $J_{HH}$  = 18Hz, 1H), 5.08 (dq,  $J_{HH}$  = 1.5 Hz,  $J_{HH}$  = 9Hz, 1H), 4.48 (q, J = 6Hz, 1H), 4.29-3.97 (m, 5H), 3.67 (m, 1H), 3.35 (d, J = 27 Hz), 1.51 (d, J = 6 Hz, 3H), 1.35-1.28 (m, 6H), 1.21 (s, 9H), 1.08 (s, 9H). Dia II. 7.74 (b, NH, 1H), 5.96-5.83 (m, 1H), 5.21(d, J = 18 Hz, 1H), 205.11(d, J = 9 Hz, 1H), 4.51 (q, J = 9Hz, 1H), 4.20-3.95 (m, 5H), 3.88 (t, J = 7.5Hz, 1H), 3.28 (d, J = 24 Hz), 1.63 (d, J = 6 Hz, 3H), 1.36-1.28 (m, 6H), 1.25 (s, 9H), 1.24 (s, 9H).

<sup>13</sup>C NMR (75.54 MHz, CDCl3): Dia I. □ 173.55 (s, CO), 134.40 (s, CH=CH<sub>2</sub>), 115.18 (s, CH=CH<sub>2</sub>), 81.76 (s, CH-ON), 68.56 (d, J = 137.48 Hz, CH-P), 62.17 (s, N-C(CH<sub>3</sub>)<sub>3</sub>), 61.56 (d, J = 6.04 Hz, CH<sub>2</sub>), 59.64 (d, J = 7.55 Hz, CH<sub>2</sub>), 41.06 (s, N-CH<sub>2</sub>), 35.36 (d, J = 5.28 Hz, CH-C(CH<sub>3</sub>)<sub>3</sub>), 29.69 (d, J = 6.04 Hz, CH-C(CH<sub>3</sub>)<sub>3</sub>), 28.15 (s, N-C(CH<sub>3</sub>)<sub>3</sub>), 19.21 (s, CH-CH<sub>3</sub>), 16.25 (d, J = 6.04 Hz, CH<sub>2</sub>CH<sub>3</sub>), 15.91 (d, J = 6.8 Hz, CH<sub>2</sub>CH<sub>3</sub>). Dia II. □ 173.42 (s, CO), 134.27 (s, CH=CH<sub>2</sub>), 116.30 (s, CH=CH<sub>2</sub>), 83.05 (s, CH-ON), 69.25 (d, J = 137.48 Hz, CH-P), 62.85 (s, N-C(CH<sub>3</sub>)<sub>3</sub>), 61.55 (d, J = 6.04 Hz, CH<sub>2</sub>), 60.04 (d, J = 7.55 Hz, CH<sub>2</sub>), 41.46 (s, N-CH<sub>2</sub>), 35.33 (d, J = 5.28 Hz, CH-C(CH<sub>3</sub>)<sub>3</sub>), 30.06 (d, J = 5.28 Hz, CH-C(CH<sub>3</sub>)<sub>3</sub>), 28.38 (s, N-C(CH<sub>3</sub>)<sub>3</sub>), 19.55 (s, CH-CH<sub>3</sub>), 16.55 (d, J = 6.80 Hz, CH<sub>2</sub>CH<sub>3</sub>), 16.30 (d, J = 6.8 Hz, CH<sub>2</sub>CH<sub>3</sub>).

## Example 5

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Synthesis of a dialkoxyamine from the alkoxyamine methylpropionic acid-SG1.

The alkoxyamine methylpropionic acid-SG1 is prepared according to Example 4.

10 g of alkoxyamine methylpropionic acid-SG1 (26 mmol) and 50 ml of dichloromethane (dried over calcium hydride) are introduced into a 250 ml reactor purged with nitrogen. 6.2 g of SOCl<sub>2</sub> (52 mmol) are added, via a dropping funnel, at room temperature. The mixture is left to react for 2 hours at room temperature with stirring and under a gentle stream of nitrogen. Evaporation under vacuum is performed to remove the excess SOCl<sub>2</sub> and the solvent. The acid chloride of the alkoxyamine is obtained, and is used in the subsequent synthesis without further purification.

The resulting oil is redissolved in 50 ml of dry dichloromethane. A mixture containing 1.2 g of 1,4-butanediol (13 mmol), 2.6 g of triethylamine (26 mmol) and 0.3 g of 4-dimethylaminopyridine (2.6 mmol) dissolved in 10 ml of dichloromethane is placed in the dropping funnel, under a nitrogen atmosphere. The above mixture is added dropwise to the reactor and the mixture is then left to react for three hours at room temperature. The reaction mixture is filtered, washed with a KHCO<sub>3</sub> solution and then washed with water.

The organic phase is recovered, dried over magnesium sulphate and evaporated to dryness under vacuum at room temperature. A solid is obtained, which is washed with cold pentane to give 5.2 g of dialkoxyamine (yield = 50%).

The dialkoxyamine was characterized by proton, carbon-13 and phosphorus NMR. <sup>31</sup>P NMR (CDCl<sub>3</sub>):  $\delta = 26$  ppm

## Example 6:

Coupling between the alkoxyamine methylpropionic acid-SG1 and a POE-Ome block  $(Mn = 750 \text{ g.mol}^{-1})$ :

$$SG_1 \longrightarrow OH + HO \left(CH_2CH_2O\right)_{\Pi}CH_3 \longrightarrow CH_2CI_2 \longrightarrow O\left(CH_2CH_2O\right)_{\Pi}CH_3$$



## United States Patent and Trademark Office

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**CONFIRMATION NO. 5047** Bib Data Sheet

SERIAL NUMBER 10/813,822	FILING DATE 03/31/2004 RULE	1	LASS É	GROL	UP ART UNIT 1626		D	ATTORNEY OCKET NO. -AM 1946 NP			
APPLICANTS											
Jean-luc Couturier, Lyon, FRANCE;											
Oliver Guerret, Mazerolles, FRANCE; Denis Bertin, Plan De Cuques, FRANCE;Didier Gigmes, Marseille, FRANCE; Sylvain Marque, Antraigues Sur Volane, FRANCE; Paul Tordo, Marseille, FRANCE; Pierre-Emmanuel Dufils, Carry Le Rouet, FRANCE;											
** CONTINUING DATA	A ************************************	*									
4	** FOREIGN APPLICATIONS ************************************										
** 06/10/2004	<u> </u>							· ·			
Foreign Priority claimed 35 USC 119 (a-d) conditions	X yes ☐ no ☐ Metafti	100	STATE OR	SHE	ETS	тот	`AL	INDEPENDENT			
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ADDRESS 31684 ARKEMA INC. PATENT DEPARTME 2000 MARKET STREI PHILADELPHIA, PA 19103-3222	ET										
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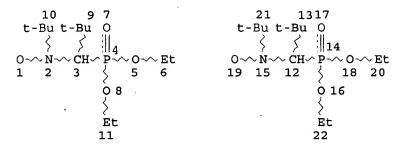
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### STEREO ATTRIBUTES: NONE

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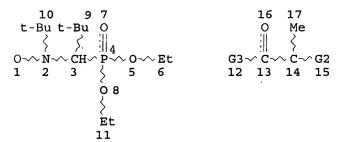
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## STEREO ATTRIBUTES: NONE



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VAR G2=H/ME

VAR G3=21/25/28/31

REP G4 = (1-5) CH2

REP G5=(1-5) 32-29 33-31

NODE ATTRIBUTES:

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DEFAULT ECLEVEL IS LIMITED

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NUMBER OF NODES IS 33
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ACCESSION NUMBER: 2006:558238 HCAPLUS

DOCUMENT NUMBER: 145:28691

TITLE: Cast plates with improved impact resistance

based on methyl methacrylate copolymers

INVENTOR(S): Guerret, Olivier; Chenard, Jean-Yves; Ederle,

Yannick

PATENT ASSIGNEE(S): Arkema, Fr.

SOURCE:

PCT Int. Appl., 57 pp. CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: French

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO:			KIN	D	DATE	TE APPLICATION NO.						DATE	
				-									
WO 20060615	23		<b>A</b> 1		2006	0615	,	WO 2	005-	FR30	87		
													2005
													1209
W: AE,	AG,	AL,	AM,	AT,	AU,	AZ,	BA,	BB,	BG,	BR,	BW,	BY,	BZ,
CA,	CH,	CN,	CO,	CR,	CU,	CZ,	DE,	DK,	DM,	DZ,	EC,	EE,	EG,
ES,	FI,	GB,	GD,	GE,	GH,	GM,	HR,	HU,	ID,	IL,	IN,	IS,	JP,
KE,	KG,	KM,	KN,	KΡ,	KR,	ΚZ,	LC,	LK,	LR,	LS,	LT,	LU,	LV,
LY,	MA,	MD,	MG,	MK,	MN,	MW,	MX,	ΜZ,	NA,	NG,	NI,	NO,	NZ,
OM,	PG,	PH,	PL,	PT,	RO,	RU,	SC,	SD,	SE,	SG,	SK,	SL,	SM,
SY,	ТJ,	TM,	TN,	TR,	TT,	TZ,	UA,	UG,	US,	UZ,	VC,	VN,	YU,
ZA,	ZM,	ZW									-		
RW: AT,	BE,	BG,	CH,	CY,	CZ,	DE,	DK,	EE,	ES,	FI,	FR,	GB,	GR,
HU,	IE,	IS,	IT,	LT,	LU,	LV,	MC,	NL,	PL,	PT,	RO,	SE,	SI,
					CG,								
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					AM,								
FR 2879205												•	
											=		2004

PRIORITY APPLN. INFO.:

FR 2004-13186

1210

2004 1210

US 2005-647056P

2005

0126

AB Me methacrylate (I) copolymers that provide cast-molded sheets with improve impact strength are manufactured by (1) polymerization of a monomer in the presence of ≥1 alkoxyamine having ≥2 groups formed from nitroxides [e.g., [Me3CCH[P(:O)(OEt)2]N(CMe3)OC HMeCO2(CH3)]2] at temps. sufficient to activate the alkoxyamine to form a core chain (glass-transition temperature <0°), (2) reaction of the core chain optionally, containing unreacted core-chain monomers with monomers destined to form branches (glass-transition temperature >0°), and (3) polymerization of I and, optionally, other monomers in the presence of the product of (2) and  $\geq 1$ radical initiator.

IT 300811-94-3

> (cast plates with improved impact resistance based on Me methacrylate copolymers manufactured in presence of multifunctional alkoxyamines)

300811-94-3 HCAPLUS RN

CN 3,7-Dioxa-4-aza-6-phosphanonanoic acid, 4,5-bis(1,1-dimethylethyl)-6-ethoxy-2-methyl-, 1,6-hexanediyl ester, 6,6'-dioxide (9CI) (CA INDEX NAME)

CC 37-3 (Plastics Manufacture and Processing)

300811-94-3

(cast plates with improved impact resistance based on Me methacrylate copolymers manufactured in presence of multifunctional alkoxyamines)

REFERENCE COUNT:

THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L26 ANSWER 2 OF 19 HCAPLUS COPYRIGHT 2006 ACS on STN

2

ACCESSION NUMBER:

2006:506 HCAPLUS

DOCUMENT NUMBER:

144:254481

TITLE:

Nitroxide-mediated radical polymerization of 2-(dimethylamino)ethyl acrylate and its sequential block copolymerization with styrene

and n-butyl acrylate

AUTHOR (S):

Bian, Kejian; Cunningham, Michael F.

CORPORATE SOURCE:

Department of Chemical Engineering, Queen's

University, Kingston, ON, K7L 3N6, Can. SOURCE:

Journal of Polymer Science, Part A: Polymer Chemistry (2005) Volume Date 2006, 44(1),

CODEN: JPACEC; ISSN: 0887-624X

John Wiley & Sons, Inc.

DOCUMENT TYPE:

Journal

PUBLISHER: LANGUAGE:

English

Nitroxide-mediated radical polymerization (NMRP) of 2-(dimethylamino)ethyl acrylate (DMAEA) was carried out at 100-120 °C, initiated by MONAMS, an alkoxyamine based on N-tert-butyl-N-(1-diethyl phosphono-2,2-dimethylpropyl)nitroxide, SG1. Controlled polymerization can be achieved by the addition of free SG1 (the initial molar ratio of SG1 to MONAMS ranged from 0.06 to 0.12), giving a linear first-order kinetic plot up to 55-70% conversion depending on the reaction conditions. The mol. wts. show a near linear increase with conversion; however, they deviate to some extent with theor. values. SG1-mediated polymerization of DMAEA at 112 °C is also controlled in organic solvents (N,N-dimethylformide, anisole, xylene). Polymerization rate increases with increasing solvent polarity. Chain transfer to polymer produces .apprx.1 mol % branches in bulk and 1.2-1.9 mol % in organic solvents, typical of those for acrylates. From poly(styrene) (pS) and poly(Bu acrylate) (pBA) macroinitiators, amphiphilic di- and triblock copolymers p(S-b-DMAEA), p(DMAEA-b-S-b-DMAEA), p(BA-b-DMAEA), and p(DMAEA-b-BA-b-DMAEA) were synthesized via NMRP at 110 °C. Polymers were characterized by GPC, NMR, surface tension measurements, and DSC.

IT 300811-94-3

> (nitroxide-mediated radical polymerization of (dimethylamino)ethyl acrylate and its sequential block copolymn. with styrene and Bu acrylate)

RN300811-94-3 HCAPLUS

3,7-Dioxa-4-aza-6-phosphanonanoic acid, 4,5-bis(1,1-dimethylethyl)-6-ethoxy-2-methyl-, 1,6-hexanediyl ester, 6,6'-dioxide (9CI) (CA INDEX NAME)

CC 35-4 (Chemistry of Synthetic High Polymers)

IT 188526-94-5 300811-93-2 **300811-94-3** 

(nitroxide-mediated radical polymerization of (dimethylamino)ethyl acrylate and its sequential block copolymn. with styrene and Bu acrylate)

REFERENCE COUNT:

40 THERE ARE 40 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L26 ANSWER 3 OF 19 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 2005:1162780 HCAPLUS

DOCUMENT NUMBER: 144:70138

TITLE: Nitroxide-Mediated Controlled Free-Radical

Emulsion Polymerization Using a Difunctional Water-Soluble Alkoxyamine Initiator. Toward the Control of Particle Size, Particle Size Distribution, and the Synthesis of Triblock

Copolymers

AUTHOR(S): Nicolas, Julien; Charleux, Bernadette;

Guerret, Olivier; Magnet, Stephanie

CORPORATE SOURCE: Laboratoire de Chimie Macromoleculaire,

Universite Pierre et Marie Curie, Paris,

75252, Fr.

SOURCE: Macromolecules (2005), 38(24), 9963-9973

CODEN: MAMOBX; ISSN: 0024-9297

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal LANGUAGE: English

A novel dialkoxyamine bearing two carboxylic acid groups was synthesized by the addition of a high dissociation rate constant alkoxyamine onto tri(ethylene glycol) diacrylate. This dialkoxyamine was first successfully used as an initiator for the bulk polymns. of Bu acrylate and styrene. Then the sodium salt counterpart was used as a water-soluble initiator in the emulsion polymns. of Bu acrylate and styrene via a multistep process. Owing to its unique structure with two carboxylate salts remaining covalently bound to the chain and hence locked at the particle surface, very stable latexes were recovered with, for the first time, small particles and narrow particle size distributions. emulsion process was successfully applied to the synthesis of well-defined poly(Bu acrylate) and polystyrene homopolymers as well as polystyrene-b-poly(Bu acrýlate)-b-polystyrene triblock copolymer. This work represents the first successful attempt of the synthesis of a complex architecture together with the control of average diameter and particle size distribution in nitroxide-mediated polymerization in emulsion, which is of high industrial and academic interest.

IT 871982-25-1P 871982-26-2P

(preparation of a difunctional water-soluble alkoxyamine initiator for nitroxide-mediated controlled free-radical emulsion polymerization)

RN 871982-25-1 HCAPLUS

PAGE 1-A

PAGE 1-B

RN 871982-26-2 HCAPLUS
CN 6,9,12,15-Tetraoxaeicosanedioic acid, 4,17-bis[[[1-(diethoxyphosphinyl)-2,2-dimethylpropyl](1,1-dimethylethyl)amino]oxy]-2,2,19,19-tetramethyl-5,16-dioxo-, disodium salt (9CI) (CA INDEX NAME)

PAGE 1-A

●2 Na

PAGE 1-B

CC 35-3 (Chemistry of Synthetic High Polymers)

IT 871982-25-1P 871982-26-2P

(preparation of a difunctional water-soluble alkoxyamine initiator for nitroxide-mediated controlled free-radical emulsion polymerization)

REFERENCE COUNT:

THERE ARE 56 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE

IN THE RE FORMAT

L26 ANSWER 4 OF 19 HCAPLUS COPYRIGHT 2006 ACS on STN

56

ACCESSION NUMBER:

2005:727462 HCAPLUS

DOCUMENT NUMBER:

144:331825

TITLE:

Use of a novel class of SG1-based water-soluble dialkoxyamine in

nitroxide-mediated controlled-free radical

emulsion polymerization

AUTHOR (S):

Nicolas, Julien; Charleux, Bernadette; Guerret, Olivier; Magnet, Stephanie

CORPORATE SOURCE:

Laboratoire de Chimie des Polymeres, CNRS -

USHA SHRESTHA EIC 1700 REM 4B28

UMR 7610 Universite Pierre et Marie Curie,

Paris, 75252/05, Fr.

SOURCE: Polymer Preprints (American Chemical Society,

Division of Polymer Chemistry) (2005), 46(2),

CODEN: ACPPAY; ISSN: 0032-3934

PUBLISHER: American Chemical Society, Division of Polymer

Chemistry

DOCUMENT TYPE: Journal; (computer optical disk)

LANGUAGE:

English A novel SG1-based water-soluble dialkoxyamine was synthesized by intermol. radical 1,2-addition of the MAMA alkoxyamine onto tri(ethylene glycol) diacrylate. It was then used as an initiator in nitroxide-mediated emulsion polymerization of n-Bu acrylate and styrene through a multi-step process. Stable latexes were recovered and all the features of a controlled system were obtained whatever the monomer. Thanks to its unique structure, the synthesis of a well-defined polystyrene-b-poly(n-Bu acrylate) -b-polystyrene triblock copolymer by sequential monomer addns. was also successfully performed.

ΙT 871982-25-1P

> (nitroxide initiator; use of a novel class of SG1-based water-soluble dialkoxyamine in nitroxide-mediated controlled-free radical emulsion polymerization)

871982-25-1 HCAPLUS RN

6,9,12,15-Tetraoxaeicosanedioic acid, 4,17-bis[[[1-CN (diethoxyphosphinyl) -2, 2-dimethylpropyl] (1, 1dimethylethyl)amino]oxy]-2,2,19,19-tetramethyl-5,16-dioxo- (9CI) (CA INDEX NAME)

PAGE 1-A

PAGE 1-B

CC 35-4 (Chemistry of Synthetic High Polymers)

IT 871982-25-1P

> (nitroxide initiator; use of a novel class of SG1-based water-soluble dialkoxyamine in nitroxide-mediated controlled-free radical emulsion polymerization)

REFERENCE COUNT:

THERE ARE 11 CITED REFERENCES AVAILABLE 11 FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

ANSWER 5 OF 19 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 2005:727381 HCAPLUS

DOCUMENT NUMBER:

CORPORATE SOURCE:

144:312375 TITLE:

One step synthesis of NMP star polymer

initiator for "core first" method

AUTHOR(S): Dufils, Pierre-Emmanuel; Gigmes, Didier;

Guerret, Olivier; Bertin, Denis; Tordo, Paul UMR-CNRS 6517 Chimie Biologie et Radicaux Libres. CNRS-Universites d'Aix-Marseille,

Marseille, 13397/20, Fr. SOURCE:

Polymer Preprints (American Chemical Society,

Division of Polymer Chemistry) (2005), 46(2),

328-329

CODEN: ACPPAY; ISSN: 0032-3934

American Chemical Society, Division of Polymer PUBLISHER:

Chemistry

DOCUMENT TYPE: Journal; (computer optical disk)

LANGUAGE: English

Star polymer synthesis can be classified in two main strategies. Firstly, the "arm first" method involves coupling reaction between a multifunctional coupling agent and preformed length controlled, end functionalized polymer chains. Secondly, the "core first" method involves the synthesis of multiple polymer chains polymerized with a multifunctional initiator prepared through multi-step syntheses. In this paper we present a "core first" strategy which overcomes the drawbacks mentioned above and provide, in one step, a NMP star polymer precursor based on nitroxide SG1 from a new alkoxyamine and com. available compds. The initiator was synthesized by the intermol. radical addition of the alkoxyamine onto a triacrylate precursor to produce a trifunctional initiator carrying three nitroxide moieties. Star polymers were obtained by nitroxide mediated polymerization of styrene with the trifunctional initiator to provide a three arm star polymer. Although the synthesis of the initiator is not optimized, the preliminary

results indicate that the polymerization of styrene is controlled. 880098-22-6P

(one step synthesis of NMP star polymer initiator)

RN 880098-22-6 HCAPLUS

IT

CN

Pentanedioic acid, 4-[[[1-(diethoxyphosphinyl)-2,2-dimethylpropyl](1,1-dimethylethyl)amino]oxy]-2,2-dimethyl-, 5,5'-[2-[4-(2-carboxy-2-methylpropyl)-6,7-bis(1,1-dimethylethyl)-8-ethoxy-8-oxido-3-oxo-2,5,9-trioxa-6-aza-8-phosphaundec-1-yl]-2-(hydroxyethyl)-1,3-propanediyl] ester (9CI) (CA INDEX NAME)

PAGE 1-A

O

EtO-P-OEt

t-Bu

N-O O HO-CH2 O Me

Me CH-C-O-CH2-C-CH2-C-CH2-C-CO2H

t-Bu-CH

HO2C-C-CH2

R

EtO-P-OEt Me

PAGE 2-A

$$\begin{array}{c|c} & \text{Me} & & & \\ & | & \\ & | & \\ & | & \\ & \text{Me} & & \\ & & CH-C-O-CH_2 \\ & & \\ & & \\ & & CH-C-O-CH_2 \\ & &$$

CC 35-3 (Chemistry of Synthetic High Polymers)

880098-22-6P

0

(one step synthesis of NMP star polymer initiator)

REFERENCE COUNT: 9 THERE ARE 9 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE

IN THE RE FORMAT

11/26 ANSWER 6 OF 19 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

2005:708387 HCAPLUS

DOCUMENT NUMBER:

143:347038

TITLE:

AUTHOR (S):

SG1 based alkoxyamines as radical initiators for the synthesis of lactones and lactams Bertin, Denis; Gigmes, Didier; Marque, Sylvain

R. A.; Tordo, Paul

CORPORATE SOURCE:

UMR 6517, Universites d'Aix-Marseille I, II et

III-CNRS, Marseille, 13397, Fr.

SOURCE:

Tetrahedron (2005), 61(36), 8752-8761

CODEN: TETRAB; ISSN: 0040-4020

PUBLISHER:

Elsevier B.V.

DOCUMENT TYPE: LANGUAGE:

Journal English

OTHER SOURCE(S):

CASREACT 143:347038

Recently, it has been shown that alkoxy amine derivs. can be used as radical initiators in tin-free radical chemical Thus, a method for the preparation of highly valuable alkoxyamines was developed, which involved ionic chemical and wherein a radical cyclization was triggered by thermal initiation. Following that procedure, bicyclic, spiro and eight-membered lactones were easily prepared in good yields with very high stereoselectivity. The reaction mechanism was discussed.

IT 763105-81-3P

(preparation of [[(tert-butyl)[methyl(oxo)propenylamino)ethoxy]amino ]dimethylpropyl]phosphonic acid ester (SG1-initiator-based alkoxyamine derivative) and study of its thermal radical cyclization)

RN 763105-81-3 HCAPLUS

CN Phosphonic acid, [1-[(1,1-dimethylethyl) [1-methyl-2-oxo-2-(2propenylamino)ethoxy]amino]-2,2-dimethylpropyl]-, diethyl ester (9CI) (CA INDEX NAME)

IT 763105-79-9P

> (preparation of di(tert-butyl)(ethoxy)-3,7-dioxa-4-aza-6phosphanonanoic acid propenyl ester oxide (SG1-initiator-based alkoxyamine derivative) and study of its thermal radical cyclization)

RN763105-79-9 HCAPLUS

3,7-Dioxa-4-aza-6-phosphanonanoic acid, 4,5-bis(1,1-dimethylethyl)-CN 6-ethoxy-2-methyl-, 2-propenyl ester, 6-oxide (9CI) (CA INDEX NAME)

$$\begin{array}{c} & \circ \\ & | | \\ & \text{EtO-P-OEt} \\ & | \\ & \text{t-Bu-N-CH-Bu-t} \\ \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & | \\ & |$$

CC 27-20 (Heterocyclic Compounds (One Hetero Atom)) Section cross-reference(s): 22

IT 763105-81-3P

(preparation of [[(tert-butyl)[methyl(oxo)propenylamino)ethoxy]amino]dimethylpropyl]phosphonic acid ester (SG1-initiator-based alkoxyamine derivative) and study of its thermal radical cyclization)

IT 763105-79-9P

(preparation of di(tert-butyl)(ethoxy)-3,7-dioxa-4-aza-6-phosphanonanoic acid propenyl ester oxide (SG1-initiator-based alkoxyamine derivative) and study of its thermal radical cyclization)

REFERENCE COUNT:

THERE ARE 48 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L26 ANSWER 7 OF 19 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

2005:547687 HCAPLUS

DOCUMENT NUMBER:

143:80747

TITLE:

Controlled radical acrylic copolymer

thickeners

INVENTOR (S):

Schmidt, Scott Charles; Callais, Peter

Anthony; Macy, Noah Eliot; Guerrett, Olivier

PATENT ASSIGNEE(S):

Arkema Inc., USA

SOURCE:

PCT Int. Appl., 40 pp.

CODEN: PIXXD2

DOCUMENT TYPE:

Patent

LANGUAGE:

English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.			KIN	D	DATE			APPL	DATE					
					-									
	-													
WO 2005	0567	39		A1 20050623				WO 2004-US34236						
														2004
														1015
W:	ΑE,	AG,	AL,	AM,	ΑT,	AU,	ΑZ,	BA,	BB,	BG,	BR,	BW,	BY,	BZ,
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	ES,	FI,	GB,	GD,	GE,	GH,	GM,	HR,	·HU,	ID,	IL,	IN,	IS,	JP,
	KE,	KG,	ΚP,	KR,	KZ,	LC,	LK,	LR,	LS,	LT,	LU,	LV,	MA,	MD,
	MG,	MK,	MN,	MW,	MX,	MZ,	NA,	NI,	NO,	NZ,	OM,	PG,	PH,	PL,
	PT,	RO,	RU,	SC,	SD,	SE,	SG,	SK,	SL,	SY,	ТJ,	TM,	TN,	TR,
	TT,	TZ,	UA,	ŪĠ,	US,	UΖ,	VC,	VN,	YU,	ZA,	ZM,	ZW		
RW:	BW,	GH,	GM,	KE,	LS,	MW,	MZ,	NA,	SD,	SL,	SZ,	TZ,	UG,	ZM,
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	CY,	CZ,	DE,	DK,	EE,	ES,	FI,	FR,	GB,	GR,	ΗU,	ΙE,	ΙΤ,	LU,
	MC,	ΝL,	PL,	PT,	RO,	SE,	SI,	SK,	TR,	BF,	ВJ,	CF,	CG,	CI,

CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

PRIORITY APPLN. INFO.:

US 2003-525549P

\_\_20.03\_\_ 1126

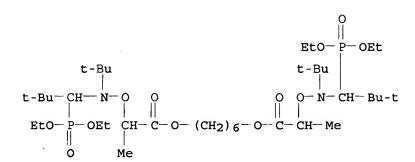
AB The present invention relates to acrylic block copolymers synthesized by a controlled radical process, and their use as thickeners in oil-based compns. The acrylic copolymers are especially useful as viscosity index improvers in lubricating oil.

IT 300811-94-3

(controlled living radical polymerized acrylic copolymer
thickeners)

RN 300811-94-3 HCAPLUS

CN 3,7-Dioxa-4-aza-6-phosphanonanoic acid, 4,5-bis(1,1-dimethylethyl)-6-ethoxy-2-methyl-, 1,6-hexanediyl ester, 6,6'-dioxide (9CI) (CA INDEX NAME)



IC ICM C10M145-14

CC 51-8 (Fossil Fuels, Derivatives, and Related Products) Section cross-reference(s): 35, 36, 66

IT 188526-94-5 300811-93-2 300811-94-3

(controlled living radical polymerized acrylic copolymer thickeners)

REFERENCE COUNT:

THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L26 ANSWER 8 OF 19 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

2005:361852 HCAPLUS

DOCUMENT NUMBER:

142:411841

TITLE:

Process for the preparation of

polyalkoxyamines for use as initiators in

radical polymerization

INVENTOR(S):

Magnet, Stephanie; Guerret, Olivier;

Couturier, Jean-Luc

PATENT ASSIGNEE(S):

Arkema, Fr.

SOURCE:

Eur. Pat. Appl., 14 pp.

CODEN: EPXXDW

DOCUMENT TYPE:

Patent

LANGUAGE:

French

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO. DATE

EP 1526138	A1	20050427	EP 2004-292480	2004
				1019
			GB, GR, IT, LI, LU, RO, MK, CY, AL, TR,	
EE, HU,		•	KO, MK, CI, AL, IK,	BG, C2,
FR 2861394	A1	20050429	FR 2003-12452	
				2003
FR 2861394	В1	20060120		1024
CA 2482501	AA		CA 2004-2482501	
				2004
WO 2005107577	2.1	20050510	170 2004 260711	1019
US 2005107577	A1	20050519	US 2004-969711	2004
				1020
CN 1629136	A	20050622	CN 2004-10095971	
				2004
JP 2005126442	A2	20050519	JP 2004-309512	1022
01 2003120412	712	20030313	01 2004 303312	2004
				1025
PRIORITY APPLN. INFO.	:		FR 2003-12452	A 2002
				2003 1024
				1024
			US 2003-514287P	P
				2003
				1024

AB Polyalkoxyamines, useful as initiators without purification from preparation mixts. for radical polymerization especially in manufacture of block polymers, are

prepared by reaction of R12C(CO2R2)ON(CMe3)CH(CMe3)P(O)(OEt)2 (R1 = C1-3 alkyl, R2 = H, C1-8 alkyl, Ph, Li, Na, K, NH4+, NBu4+, or NHBu3+) (I) with Z(CH:CH2)n [Z = aryl or Z1(XCO)n; Z1 = polyfunctional compound such as polyol, X = O, N having a C-containing group, or H, or S;  $n \geq 2$ ] (II) optionally in a solvent at 0-90° and I-II mol ratio n-1.5n. A typical polyalkoxyamine was manufactured by reaction of 42.1 g 2-bromo-2-methylpropionic acid 90 min with 78.9 g (EtO)2P(O)CH(CMe3)N(CMe3)O $\bullet$  in PhMe in the presence of Cu, CuBr, and N,N,N',N',N''- pentamethyldiethylenetriamine and reaction of 2 g resulting monoalkoxyamine 20 h at reflux with 0.55 g 1,4-butanediol diacrylate in EtOH.

IT 850348-10-6P

(preparation of polyalkoxyamines for use as initiators in radical polymerization)

RN 850348-10-6 HCAPLUS

CN Pentanedioic acid, 4-[[[1-(diethoxyphosphinyl)-2,2-dimethylpropyl](1,1-dimethylethyl)amino]oxy]-2,2-dimethyl-, 5,5'-(1,4-butanediyl) ester (9CI) (CA INDEX NAME)

IC ICM C07F009-40 ICS C08G073-00

35-4 (Chemistry of Synthetic High Polymers) CC

Section cross-reference(s): 29

IT 850348-10-6P

(preparation of polyalkoxyamines for use as initiators in radical

polymerization)

REFERENCE COUNT:

THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

ACCESSION NUMBER:

L26 ANSWER 9 OF 19 HCAPLUS COPYRIGHT 2006 ACS on STN 2005:142258 HCAPLUS

DOCUMENT NUMBER:

142:374222

TITLE:

Surface initiated polymerization of poly(butyl

acrylate) by nitroxide mediated polymerization: first comparative polymerization of a bimolecular and a unimolecular initiator-grafted silica

particles

AUTHOR (S):

Parvole, Julien; Laruelle, Gael; Khoukh,

Abdel; Billon, Laurent

CORPORATE SOURCE:

Laboratoire de Physico-Chimie des Polymeres, UMR 5067 CNRS, Universite de Pau et Pays de l'Adour Helioparc Pau-Pyrenees, Pau, 64053,

SOURCE:

Macromolecular Chemistry and Physics (2005),

206(3), 372-382

CODEN: MCHPES; ISSN: 1022-1352

PUBLISHER:

Wiley-VCH Verlag GmbH & Co. KGaA

DOCUMENT TYPE:

Journal

LANGUAGE:

English

Hybrid silica-particles comprising an inorg. core and an organic polymer shell were synthesized by nitroxide mediated polymerization of Bu acrylate in the presence of a grafted alkoxyamine. The layer of initiator mols. attached to the surface of silica is based on an acyclic β-phosphonylated nitroxide also referred to SG1. The synthesis and the characterization by NMR of this alkoxyamine structure are briefly described. Moreover, the grafted initiator particles were studied by XPS, NMR 29Si CP/MAS, and TGA to determine the nature of the anchorage sites and the grafting d. With this stable radical as chain growth moderator tethered to the inorg. core, the grafting from polymerization exhibits a control character with

a low polydispersity index (I < 1.1). For the first time, a comparative study with a bimol. system based on a grafted azoic initiator in presence of SG1 is also described. A different kinetic behavior was demonstrated and interpreted in terms of variation of the initial [monomer]/[initiator] ratio.

IT 763105-79-9P

(synthesis of allyl alkoxyamine for surface initiated graft polymerization of poly(Bu acrylate) on silica particles)

RN 763105-79-9 HCAPLUS

CN 3,7-Dioxa-4-aza-6-phosphanonanoic acid, 4,5-bis(1,1-dimethylethyl)-6-ethoxy-2-methyl-, 2-propenyl ester, 6-oxide (9CI) (CA INDEX NAME)

CC 35-4 (Chemistry of Synthetic High Polymers)
 Section cross-reference(s): 38

IT 763105-79-9P

(synthesis of allyl alkoxyamine for surface initiated graft polymerization of poly(Bu acrylate) on silica particles)

REFERENCE COUNT:

THERE ARE 52 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L26 ANSWER 10 OF 19 HCAPLUS COPYRIGHT 2006 ACS on STN

52

ACCESSION NUMBER:

2004:822885 HCAPLUS

DOCUMENT NUMBER:

141:296170

TITLE:

 $\beta$ -Phosphorylated alkoxyamines and their

use in preparing  $\alpha-\omega$ 

functionalized monomers or polymers

INVENTOR(S):

Couturier, Jean-Luc; Guerret, Olivier

PATENT ASSIGNEE(S):

Atofina, Fr.

SOURCE:

Eur. Pat. Appl., 15 pp.

CODEN: EPXXDW

DOCUMENT TYPE:

Patent

LANGUAGE:

French

A1

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

FR 2853317

PATENT NO.			KIND DATE			APPLICATION NO.					DATE		
EP 1464	- 648			A1		2004	1006	EP 2	004-	2907	77		2004 0323
R:	MC,	PT,	IE,	-	LT,		•	GR, MK,	•	•	•	-	-

20041008

2003

FR 2003-3999

					0401
FR 2853317	B1	20060707			
CA 2462923	AA	20041001	CA 2004-2462923		
					2004
					0330
US 2005065119	A1	20050324	US 2004-813822		
					2004
					0331
JP 2004307502	A2	20041104	JP 2004-109037		
					2004
					0401
CN 1576280	Α	20050209	CN 2004-10038709		
					2004
					0401
PRIORITY APPLN. INFO.:			FR 2003-3999	Α	
					2003
					0401

 $\leq M$ 

OTHER SOURCE(S): MARPAT 141:296170

β-Phosphorylated alkoxyamines A-OCCMe(R)(M)nON(CMe3)CH(CMe3)P (O) (OEt) 2 [I; A = HO, M1O, R1O, Cl; M1 = Li, Na, K, H4N+, Bu4N+, Bu3NH+; R = H, Me; R1 = (un)branched C1-6 alkyl; M = monomeric, vinylic chain susceptible to radical polymerization, preferably (un) substituted styrene, dienes, (meth) acrylic acids, alkyl (meth) acrylates, acrylonitrile, acrylamide derivs., vinylpyrrolidinone or a mixture of at least 2 of these; n = an integer which can be 0], preferably I (A = HO, Cl; n = 0), are claimed for use in preparation of mono- or polyalkoxyamines Z[OCCMe(R)(M)nON(CMe3)CH(CMe3)P(O)(OEt)2]x[II; same R, M, n; x]≥ 1; Z = mono- or polyfunctional structure, e.g., CH2:CHCH2O, CH2:CHCH2NH, Me(OCH2CH2)pO, O(CH2)qO; p,  $q = \ge$ 1), preferably II (Z = CH2:CHCH2O, CH2:CHCH2NH, n = 0, R = H, x =1; Z = O(CH2)4O, n = 0, R = Me, x = 2; Z = Me(OCH2CH2)pO, n = 0, R= Me, x = 1). In an example, treating 5.4 mmol HO2CCHMeON(CMe3)CH(CMe3)P(O)(OEt)2 (preparation given) in 25 mL CH2Cl2 with 16.2 mmol SOC12 45 min at room temperature gave the corresponding acid chloride, which was subsequently dissolved in 30 mL Et2O and treated with 10.8 mmol allyl alc., 5.4 mmol Et3N, 1.1 mmol DMAP and 10 mL Et20 for 2 h at room temperature to afford, after workup, 60% CH2:CHCH2O2CCHMeON(CMe3)CH(CMe3)P(O)(OEt)2.

TT 763105-79-9P 763105-81-3P 763105-82-4P 763105-83-5P

(preparation of  $\alpha$ - $\omega$  functionalized monomers or polymers from  $\beta$ -phosphorylated alkoxyamines) 763105-79-9 HCAPLUS

CN 3,7-Dioxa-4-aza-6-phosphanonanoic acid, 4,5-bis(1,1-dimethylethyl)-6-ethoxy-2-methyl-, 2-propenyl ester, 6-oxide (9CI) (CA INDEX NAME)

RN

RN 763105-81-3 HCAPLUS

CN Phosphonic acid, [1-[(1,1-dimethylethyl)[1-methyl-2-oxo-2-(2-propenylamino)ethoxy]amino]-2,2-dimethylpropyl]-, diethyl ester (9CI) (CA INDEX NAME)

$$\begin{array}{c} & \text{O} \\ || \\ \text{EtO-P-OEt} \\ | \\ \text{t-Bu-N-CH-Bu-t} \\ || \\ \text{H}_2\text{C} = \text{CH-CH}_2\text{-NH-C-CH-O} \\ || &| \\ \text{O Me} \end{array}$$

RN 763105-82-4 HCAPLUS

CN 3,7-Dioxa-4-aza-6-phosphanonanoic acid, 4,5-bis(1,1-dimethylethyl)-6-ethoxy-2,2-dimethyl-, 1,4-butanediyl ester, 6,6'-dioxide (9CI) (CA INDEX NAME)

RN 763105-83-5 HCAPLUS

CN Poly(oxy-1,2-ethanediyl),  $\alpha$ -[4,5-bis(1,1-dimethylethyl)-6-ethoxy-2,2-dimethyl-6-oxido-1-oxo-3,7-dioxa-4-aza-6-phosphanon-1-yl]- $\omega$ -methoxy- (9CI) (CA INDEX NAME)

IC ICM C07F009-40

ICS C08F004-00

CC 29-7 (Organometallic and Organometalloidal Compounds)
Section cross-reference(s): 35

763105-79-9P 763105-81-3P 763105-82-4P 763105-83-5P

(preparation of  $\alpha\text{-}\omega$  functionalized monomers or polymers from  $\beta\text{-phosphorylated}$  alkoxyamines)

L26 ANSWER 11 OF 19 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

2003:591230 HCAPLUS

DOCUMENT NUMBER:

İΤ

139:134362

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TITLE:
                         Method of producing and using materials which
                         are reinforced against impact and which
                         contain block copolymers that are obtained by
                         means of controlled radical polymerization in
                         the presence of nitroxides
INVENTOR(S):
                         Ruzette, Anne-valerie; Chauvin, Florence;
                         Guerret, Olivier; Bertin, Denis; Vuillemin,
                         Bruno; Leibler, Ludwik; Gerard, Pierre;
                         Ederle, Yannick
PATENT ASSIGNEE(S):
                         ATOFINA, Fr.
SOURCE:
                         PCT Int. Appl., 30 pp.
                         CODEN: PIXXD2
DOCUMENT TYPE:
                         Patent
LANGUAGE:
                         French
FAMILY ACC. NUM. COUNT:
PATENT INFORMATION:
     PATENT NO.
                         KIND
                                DATE
                                           APPLICATION NO.
                                                                    DATE
     WO 2003062293
                         A1
                                20030731
                                            WO 2003-FR186
                                                                    2003
                                                                    0121
     WO 2003062293
                          C1
                                20050428
             AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA,
             CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI,
             GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG,
             KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK,
             MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SC, SD,
             SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ,
             VC, VN, YU, ZA, ZM, ZW
         RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM,
             AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ,
             DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL,
             PT, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ,
             GW, ML, MR, NE, SN, TD, TG
     CA 2473791
                          AA
                                20030731
                                            CA 2003-2473791
                                                                    2003
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     EP 1468029
                          A1
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                                            EP 2003-712271
                                                                    2003
                                                                    0121
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             MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ,
             EE, HU, SK
     JP 2005515281
                          T2
                                20050526
                                            JP 2003-562170
                                                                    2003
                                                                    0121
     CN 1643013
                          Α
                                20050720
                                            CN 2003-805719
                                                                    2003
                                                                    0121
     US 2006063891
                          A1
                                20060323
                                            US 2005-502216
                                                                    2005
                                                                    1116
PRIORITY APPLN. INFO.:
                                            FR 2002-814
                                                                    2002
                                                                    0122
                                            FR 2002-1765
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2002

0213

WO 2003-FR186

2003

0121

AB The invention relates to the production and use of block copolymers which are obtained by means of controlled radical polymerization in the presence of nitroxides for the purpose of reinforcing brittle polymer matrixes. The invention offers advantages such as (i) simplicity of copolymer synthesis and use and (ii) fine dispersion of the copolymer mols. in the brittle matrix, which ensures both the transparency of the material and high reinforcement against impact. More specifically, the invention relates to the radical synthesis of block copolymers comprising at least three blocks, which include one block having a glass transition temperature of less than 0°C and a thermoplastic end block having a glass transition temperature of more than 0°C, thereby quaranteeing compatibility with the brittle matrix to be reinforced against impact. A typical block copolymer was manufactured by radical polymerization of 3600 g Bu acrylate at 115° in the presence of 59.7073 g CH2[(CH2)3OCOCHMeON(CMe3)CH(CMe3)P(:0)(OEt)2]2 and 3.1907 q (EtO)2P(:O)CH(CMe3)N(CMe3)O→, and polymerization of 6250 g Me methacrylate at 120° in the presence of 1800 g resulting intermediate polymer.

IT 300811-94-3 300811-95-4

(manufacture of block copolymer impact improvers by controlled radical polymerization in presence of nitroxides and alkoxyamines)

RN 300811-94-3 HCAPLUS

CN 3,7-Dioxa-4-aza-6-phosphanonanoic acid, 4,5-bis(1,1-dimethylethyl)-6-ethoxy-2-methyl-, 1,6-hexanediyl ester, 6,6'-dioxide (9CI) (CA INDEX NAME)

RN 300811-95-4 HCAPLUS

CN 3,7-Dioxa-4-aza-6-phosphanonanoic acid, 4,5-bis(1,1-dimethylethyl)-6-ethoxy-2-methyl-, (2,4,6-trioxo-1,3,5-triazine-1,3,5(2H,4H,6H)-triyl)tri-2,1-ethanediyl ester, 6,6',6'!-trioxide (9CI) (CA INDEX NAME)

PAGE 1-A

PAGE 1-B

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OEt
 -CH-Bu-t
     ICM C08F293-00
IC
     ICS C08L053-00; C08L101-00
     37-6 (Plastics Manufacture and Processing)
CC
     Section cross-reference(s): 35
IT
     30Ø811-94-3 300811-95-4
        (manufacture of block copolymer impact improvers by controlled
       radical polymerization in presence of nitroxides and alkoxyamines)
REFERENCE COUNT:
                               THERE ARE 7 CITED REFERENCES AVAILABLE
                         7
                               FOR THIS RECORD. ALL CITATIONS AVAILABLE
                               IN THE RE FORMAT
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L26 ANSWER 12 OF 19 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 2

2002:709890 HCAPLUS

DOCUMENT NUMBER:

137:353388

TITLE:

Controlled/Living Radical Polymerization of tert-Butyl Acrylate Mediated by Chiral

Nitraridas A Characabanical Chudu

Nitroxides. A Stereochemical Study

AUTHOR(S):

Ananchenko, Gennady; Matyjaszewski, Krzysztof Department of Chemistry, Carnegie Mellon

University, Pittsburgh, PA, 15213, USA

SOURCE:

Macromolecules (2002), 35(22), 8323-8329

CODEN: MAMOBX; ISSN: 0024-9297

PUBLISHER:

American Chemical Society

DOCUMENT TYPE:

Journal

LANGUAGE:

CORPORATE SOURCE:

English

AB The two diastereomeric alkoxyamines of tBP-DEPN, where tBP is 1-(tert-butoxycarbonyl)ethyl and DEPN is N-(2-methylpropyl)-N-(1-diethylphosphophono-2,2-dimethylpropyl)aminoxyl, have marked

differences in their thermodn. stability (ratio of diastereomers is 5:1 at 100° in o-dichlorobenzene). They were used as initiators for the controlled/living radical polymerization of tert-Bu acrylate to test the premise that such moderators could potentially affect the tacticity of the resulting poly(tert-Bu acrylate). 2D NMR was used to analyze the end group configuration for the samples with shorter chain lengths (DP = 15-20). Although the diastereomeric excess in the polymer alkoxyamine end group is even higher than for the model compound (ratio of diastereomers is 7:1), the distribution of terminal triads in poly(tert-Bu acrylate) does not differ from those in the entire chain and is identical to that of the polymers prepared by ATRP (atom transfer radical polymerization). Thus, the tacticities of the poly(tert-Bu acrylate)s prepared by DEPN-mediated polymerization, ATRP, and conventional free radical polymerization were the same.

TΤ 474832-44-5P

CN

(initiator; preparation of chiral nitroxide initiators and stereochem. study on controlled/living radical polymerization of tert-Bu acrylate mediated by chiral nitroxides)

RN474832-44-5 HCAPLUS

> Heptanedioic acid, 2,6-bis[[[1-(diethoxyphosphinyl)-2,2dimethylpropyl](1,1-dimethylethyl)amino]oxy]-, dimethyl ester (9CI) (CA INDEX NAME)

CC 35-3 (Chemistry of Synthetic High Polymers) IT

474832-43-4P 474832-44-5P (initiator; preparation of chiral nitroxide initiators and stereochem. study on controlled/living radical polymerization of

tert-Bu acrylate mediated by chiral nitroxides)

41 THERE ARE 41 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE

IN THE RE FORMAT

ANSWER 13 OF 19 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

RÉFERENCE COUNT:

2002:624966 HCAPLUS

DOCUMENT NUMBER:

137:353359

TITLE:

Use of a difunctional alkoxyamine initiator in the miniemulsion polymerization of n-butyl

acrylate

AUTHOR (S):

Farcet, Celine; Charleux, Bernadette; Pirri,

Rosangela; Guerret, Olivier

CORPORATE SOURCE:

Lab. Chimie Macormoleculaire, UMR 760, Univ.

Pierre et Marie Curie, Paris, 75252, Fr.

SOURCE:

Polymer Preprints (American Chemical Society, Division of Polymer Chemistry) (2002), 43(2),

98-99

CODEN: ACPPAY; ISSN: 0032-3934

PUBLISHER: American Chemical Society, Division of Polymer

Chemistry

DOCUMENT TYPE: Journal; (computer optical disk)

LANGUAGE: English

AB The application of controlled free-radical polymerization to aqueous dispersed systems offers the opportunity to directly prepare well-defined copolymers in a latex form. In the presented work a SG1-based difunctional alkoxyamine was used as an initiator for the homopolymn. of Bu acrylate in aqueous miniemulsion, first to increase the achievable molar mass and second, to use the polymer as a difunctional macroinitiator for the synthesis of triblock copolymers in aqueous dispersed systems. Well-defined polymers with one alkoxyamine functionality at each end were obtained, providing that monomer conversion was kept below 70%. Beyond this conversion, extensive broadening of the molar mass distribution was evidenced, as the consequence of termination and transfer to polymer. These  $\alpha, \omega$ -difunctional homopolymers were used as precursors for the synthesis of triblock copolymers.

IT 300811-94-3

(difunctional alkoxyamine initiator in miniemulsion polymerization of Bu acrylate)

RN 300811-94-3 HCAPLUS

CN 3,7-Dioxa-4-aza-6-phosphanonanoic acid, 4,5-bis(1,1-dimethylethyl)-6-ethoxy-2-methyl-, 1,6-hexanediyl ester, 6,6'-dioxide (9CI) (CA INDEX NAME)

CC 35-3 (Chemistry of Synthetic High Polymers)

IT 300811-94-3

(difunctional alkoxyamine initiator in miniemulsion polymerization of Bu acrylate)

REFERENCE COUNT:

18 THERE ARE 18 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L26 ANSWER 14 OF 19 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

2002:319042 HCAPLUS

DOCUMENT NUMBER:

137:20639

TITLE:

Synthesis and Characterization of

Poly(styrene-b-n-butyl acrylate-b-styrene)
Triblock Copolymers Using a Dialkoxyamine as

Initiator

AUTHOR(S):

Robin, Sophie; Guerret, Olivier; Couturier,

Jean-Luc; Pirri, Rosangela; Gnanou, Yves

CORPORATE SOURCE: Laboratoire de Chimie des Polymeres

Organiques, ENSCPB-CNRS-Universite Bordeaux 1,

Talence, 33402, Fr.

SOURCE: Macromolecules (2002), 35(10), 3844-3848

CODEN: MAMOBX; ISSN: 0024-9297

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal LANGUAGE: English

This study investigates the ability of a novel difunctional alkoxyamine based on N-tert-butyl-1-diethylphosphono-2,2-dimethylpropyl nitroxide (SG1) to serve as initiator for the controlled radical polymerization of styrene (S) and Bu acrylate (nBuA). The efficiency of this initiator was checked using three different methods. After we set up the conditions best suited to the synthesis of perfectly difunctional poly(Bu acrylate) (PnBuA) samples, well-defined poly(styrene-b-Bu acrylate-b-styrene) triblock copolymers could be obtained by sequential polymerization of the corresponding monomers. However, a loss of control of the targeted structure was observed whenever the conversion of styrene exceeded 40%.

## IT 300811-94-3

(initiators; preparation and characterization of Bu acrylate-styrene triblock copolymers using dialkoxyamine as initiator)

RN 300811-94-3 HCAPLUS

CN 3,7-Dioxa-4-aza-6-phosphanonanoic acid, 4,5-bis(1,1-dimethylethyl)-6-ethoxy-2-methyl-, 1,6-hexanediyl ester, 6,6'-dioxide (9CI) (CA INDEX NAME)

CC 35-3 (Chemistry of Synthetic High Polymers)

IT 300811-94-3

(initiators; preparation and characterization of Bu acrylate-styrene triblock copolymers using dialkoxyamine as initiator)

REFERENCE COUNT:

THERE ARE 21 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L26 ANSWER 15 OF 19 HCAPLUS COPYRIGHT 2006 ACS on STN

21

ACCESSION NUMBER:

2002:151910 HCAPLUS

DOCUMENT NUMBER:

136:325922

TITLE:

Synthesis of Stars and Starlike Block

Copolymers from a Trialkoxyamine Used as

Initiator

AUTHOR(S):

Robin, Sophie; Guerret, Olivier; Couturier,

Jean-Luc; Gnanou, Yves

CORPORATE SOURCE:

Laboratoire de Chimie des Polymeres

Organiques, ENSCPB-CNRS-Universite Bordeaux-I,

Talence, 33402, Fr.

SOURCE: Macromolecules (2002), 35(7), 2481-2486

CODEN: MAMOBX; ISSN: 0024-9297

PUBLISHER:

American Chemical Society

DOCUMENT TYPE:

Journal

LANGUAGE:

English

AB This study investigates the efficiency of a novel trifunctional alkoxyamine (1) based on N-tert-butyl-1-diethylphosphono-2,2-dimethylpropyl nitroxide (SG1) (2) as initiator for the controlled radical polymerization of styrene (S) and Bu acrylate (nBuA). Three factors, namely the concentration of SG1 initially introduced in excess, the monomer conversion, and-in the specific case of PS samples-the monomer autopolymn., were found to control the quality of the star samples obtained. Well-defined T-(PBuA-b-PS)3 star block copolymers could also be synthesized by sequential copolymn. using the same trifunctional initiator.

IT 300811-95-4P

(catalyst; in synthesis of stars and starlike block copolymers from a trialkoxyamine used as initiator)

RN 300811-95-4 HCAPLUS

CN 3,7-Dioxa-4-aza-6-phosphanonanoic acid, 4,5-bis(1,1-dimethylethyl)-6-ethoxy-2-methyl-, (2,4,6-trioxo-1,3,5-triazine-1,3,5(2H,4H,6H)-triyl)tri-2,1-ethanediyl ester, 6,6',6''-trioxide (9CI) (CA INDEX NAME)

PAGE 1-A

PAGE 1-B

CC 35-4 (Chemistry of Synthetic High Polymers)

IT 300811-95-4P

(catalyst; in synthesis of stars and starlike block copolymers from a trialkoxyamine used as initiator)

REFERENCE COUNT:

26 THERE ARE 26 CITED REFERENCES AVAILABLE

# FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L26 ANSWER 16 OF 19 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

2001:747176 HCAPLUS

DOCUMENT NUMBER:

135:289208

TITLE:

Multimodal polymers by controlled radical

polymerization in the presence of alkoxyamines

Guerret, Olivier; Robin, Sophie; Gnanou, Yves

INVENTOR(S):
PATENT ASSIGNEE(S):

ATOFINA, Fr.

SOURCE:

Eur. Pat. Appl., 24 pp.

CODEN: EPXXDW

DOCUMENT TYPE:

Patent

LANGUAGE:

French ·

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1142913	A1	20011010	EP 2001-106802	2001
MC, PT, IE,	SI, LT	, LV, FI, RO		0319 SE,
FR 2807439	A1	20011012	FR 2000-4557	2000 0407
FR 2807439 CA 2343339	B1 AA	20030613 20011007	CA 2001-2343339	
CA 2343339	С	20011007		2001 0406
CN 1318570	A	20011024	CN 2001-117832	2001
JP 2001316409	A2	20011113	JP 2001-109720	0407 2001
US 2002040117	A1	20020404	US 2001-828476	0409
US 6646079	В2	20031111		2001 0409
PRIORITY APPLN. INFO.:			FR 2000-4557	A 2000 0407

GΙ

- I,  $R=(CH_2)_2O_2CCHMeON(CMe_3)CH(CMe_3)P(O)(OEt)_2$
- AB Polymers with multimodal d.p. are manufactured by radical polymerization of monomers in the presence of alkoxyamines having ≥1 amine group and groups with mol. weight >15 in the β-position to the N, such as triazine derivative I.
- CN 3,7-Dioxa-4-aza-6-phosphanonanoic acid, 4,5-bis(1,1-dimethylethyl)-6-ethoxy-2-methyl-, 1,6-hexanediyl ester, 6,6'-dioxide (9CI) (CA INDEX NAME)

- RN 300811-95-4 HCAPLUS
- CN 3,7-Dioxa-4-aza-6-phosphanonanoic acid, 4,5-bis(1,1-dimethylethyl)-6-ethoxy-2-methyl-, (2,4,6-trioxo-1,3,5-triazine-1,3,5(2H,4H,6H)-triyl)tri-2,1-ethanediyl ester, 6,6',6''-trioxide (9CI) (CA INDEX NAME)

#### PAGE 1-A

PAGE 1-B

IC ICM C08F004-00

ICS C08F293-00; C08F002-38

CC 35-4 (Chemistry of Synthetic High Polymers)

IT 78-67-1, Azobisisobutyronitrile 300811-93-2 300811-94-3

**300811-95-4** 364731-73-7

(multimodal polymers by controlled radical polymerization in presence

IN THE RE FORMAT

HCAPLUS COPYRIGHT 2006 ACS on STN

of alkoxyamines)

V

ACCESSION NUMBER:

L26 ANSWER 17 OF 19

REFERENCE COUNT:

2001:31452 HCAPLUS

DOCUMENT NUMBER:

134:101275

TITLE:

Preparation of mono and multifunctional alkoxyamines for forming nitroxyl radical initiators and regulators useful in the preparation of polymers with narrow

THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE

polydispersity

INVENTOR(S):

Kramer, Andreas; Nesvadba, Peter; Zink,

Marie-Odile; Wunderlich, Wiebke

PATENT ASSIGNEE(S):

Ciba Specialty Chemicals Holding Inc., Switz.

SOURCE: PCT Int. Appl., 74 pp.

CODEN: PIXXD2

DOCUMENT TYPE:

Patent

LANGUAGE:

English

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

PATENT NO.	KIND DATE	APPLICATION NO.	DATE
WO 2001002345	A2 20010111	WO 2000-EP5899	2000 0626
CN, CR, CI GH, GM, HI LC, LK, LI MZ, NO, NI TM, TR, T' BY, KG, KI RW: GH, GM, KI CH, CY, DI	J, CZ, DE, DK, DM, R, HU, ID, IL, IN, R, LS, LT, LU, LV, Z, PL, PT, RO, RU, T, TZ, UA, UG, US, Z, MD, RU, TJ, TM E, LS, MW, MZ, SD, E, DK, ES, FI, FR,	BA, BB, BG, BR, BY, CA DZ, EE, ES, FI, GB, GD IS, JP, KE, KG, KP, KR MA, MD, MG, MK, MN, MW SD, SE, SG, SI, SK, SL UZ, VN, YU, ZA, ZW, AM SL, SZ, TZ, UG, ZW, AT GB, GR, IE, IT, LU, MC CM, GA, GN, GW, ML, MR	, CH, , GE, , KZ, , MX, , TJ, , AZ,
SN, TD, TO CA 2375806		CA 2000-2375806	2000
EP 1189875	A2 20020327	EP 2000-951302	0626 2000
R: AT, BE, C	B1 20040804 H, DE, DK, ES, FR, I, LT, LV, FI, RO	GB, GR, IT, LI, LU, NL	0626 , MC,
JP 2003503474		JP 2001-507787	2000 · 0626
AT 272610	E 20040815	AT 2000-951302	2000 0626
US 6875831	B1 20050405	US 2001-19618	2001 1220 \
CIORITY APPLN. INFO.:		EP 1999-810567	A 1999 0702
		WO 2000-EP5899	W 2000 0626

OTHER SOURCE(S): MARPAT 134:101275

IT 319458-54-3P

(initiator; preparation of mono and multifunctional alkoxyamines as initiators for free radical polymerization with narrow polydispersity)

RN 319458-54-3 HCAPLUS

CN Octanedioic acid, bis[1-[4-[3,4-bis(1,1-dimethylethyl)-5-ethoxy-1methyl-5-oxido-2,6-dioxa-3-aza-5-phosphaoct-1-yl]phenyl]ethyl]
ester (9CI) (CA INDEX NAME)

AB The title alkoxyamines especially useful for the living polymerization of unsatd. monomers or/and oligomers giving polymers with good conversion are compds. bearing groups which can liberate stable free nitroxyl radicals of specific structures.

PAGE 1-A

```
t-Bu
 t-Bu-CH-
          -- N-
         OEt Me-CH
                                                                 CH-
                                                    Me
       0
                                      - (CH<sub>2</sub>)<sub>6</sub>
                                              - C-- O-- CH
                                                              PAGE 1-B
   Eto-P-OEt
  t-Bu
     N-CH-Bu-t
     - Me
IC
     ICM C07C239-20
     ICS C07D211-94; C08F004-00
CC
     35-3 (Chemistry of Synthetic High Polymers)
IT
     264280-51-5P
                     319457-98-2P
                                     319457-99-3P
                                                     319458-00-9P
     319458-01-0P
                     319458-02-1P
                                     319458-03-2P
                                                     319458-05-4P
     319458-06-5P
                     319458-07-6P
                                     319458-09-8P
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     319458-13-4P
                     319458-14-5P
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     319458-20-3P
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     319458-34-9P
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                                     319458-51-0P 319458-54-3P
        (initiator; preparation of mono and multifunctional alkoxyamines as
        initiators for free radical polymerization with narrow polydispersity)
L26
    ANSWER 18 OF 19 HCAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER:
                          2000:842098 HCAPLUS
DOCUMENT NUMBER:
                          134:29789
TITLE:
                          Alkoxyamine phosphonates and their use as
                          polymerization catalysts
INVENTOR (S):
                          Guerret, Olivier; Couturier, Jean-Luc; Lutz,
                          Jean-Francois; Le Mercier, Christophe; Robin,
                          Sophie; Vuillemin, Bruno
PATENT ASSIGNEE(S):
                          ATOFINA, Fr.
                          PCT Int. Appl., 59 pp.
SOURCE:
```

PATENT NO. KIND DATE APPLICATION NO. DATE

CODEN: PIXXD2

Patent

French

DOCUMENT TYPE:

FAMILY ACC. NUM. COUNT: PATENT INFORMATION:

LANGUAGE:

USHA SHRESTHA EIC 1700 REM 4B28

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     WO 2000071501
                          A1
                                 20001130
                                             WO 2000-FR1287
                                                                     2000
                                                                     0512
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             CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM,
             HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK,
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             PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT,
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             MD, RU, TJ, TM
         RW: GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH,
             CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT,
             SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN,
             TD, TG
     FR 2794459
                                 20001208
                                             FR 1999-6329
                          Α1
                                                                     1999
                                                                     0519
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                                 20040903
                          B1
     EP 1178955
                          A1
                                 20020213
                                             EP 2000-929608
                                                                     2000
                                                                     0512
             AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE,
             MC, PT, IE, SI, LT, LV, FI, RO
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                                 20030107
                                             JP 2000-619758
     JP 2003500378
                                                                     2000
                                                                     0512
     US 6657043
                          B1
                                 20031202
                                             US 2002-979124
                                                                     2002
                                                                     0315
PRIORITY APPLN. INFO.:
                                             FR 1999-6329
                                                                     1999
                                                                     0519
                                             WO 2000-FR1287
                                                                     2000
                                                                     0512
AB
     Alkoxyamine phosphonates containing 2-3 alkoxyamine groups are
     obtained from β-substituted nitroxides such as di-Et
     2,2-dimethyl-1-(tert-butylamino)propylphosphonate N-oxide (I) and
     are suitable for application as radical polymerization catalysts with
     good control. The inventive compds. can be used as initiators for
     (co)polymns. of at least one radically polymerizable monomer.
     Thus, I was condensed (2:1) with the Br groups in 1,6-hexanediol
     bis (2-bromopropionate) to give a bis (alkoxyamine phosphonate)
     which could be used to homopolymerize styrene or block
     copolymerize styrene with Bu acrylate.
IT
     300811-94-3P 300811-95-4P 310878-79-6P
     310878-80-9P 310878-81-0P 310878-82-1P
     310878-83-2P
        (production of alkoxyamine phosphonates for use as radical polymerization
        catalysts)
RN
     300811-94-3 HCAPLUS
```

3,7-Dioxa-4-aza-6-phosphanonanoic acid, 4,5-bis(1,1-dimethylethyl)-6-ethoxy-2-methyl-, 1,6-hexanediyl ester, 6,6'-dioxide (9CI) (CA

CN

INDEX NAME)

RN 300811-95-4 HCAPLUS

CN 3,7-Dioxa-4-aza-6-phosphanonanoic acid, 4,5-bis(1,1-dimethylethyl)-6-ethoxy-2-methyl-, (2,4,6-trioxo-1,3,5-triazine-1,3,5(2H,4H,6H)-triyl)tri-2,1-ethanediyl ester, 6,6',6''-trioxide (9CI) (CA INDEX NAME)

PAGE 1-A

PAGE 1-B

RN 310878-79-6 HCAPLUS

CN Hexanedioic acid, bis[4,5-bis(1,1-dimethylethyl)-6-ethoxy-6-oxido-2-phenyl-3,7-dioxa-4-aza-6-phosphanon-1-yl] ester (9CI) (CA INDEX NAME)

RN 310878-80-9 HCAPLUS

CN Butanedioic acid, bis[4,5-bis(1,1-dimethylethyl)-6-ethoxy-6-oxido-2-phenyl-3,7-dioxa-4-aza-6-phosphanon-1-yl] ester (9CI) (CA INDEX NAME)

RN 310878-81-0 HCAPLUS

CN Phosphonic acid, [phosphinidynetris[oxy(1-phenyl-2,1ethanediyl)oxy[(1,1-dimethylethyl)imino](2,2dimethylpropylidene)]]tris-, hexaethyl ester (9CI) (CA INDEX
NAME)

RN 310878-82-1 HCAPLUS

CN Phosphonic acid, [1,4-phenylenebis[ethylideneoxy[(1,1-dimethylethyl)imino](2,2-dimethylpropylidene)]]bis-, tetraethyl ester (9CI) (CA INDEX NAME)

RN 310878-83-2 HCAPLUS

CN 3,7-Dioxa-4-aza-6-phosphanonanoic acid, 4,5-bis(1,1-dimethylethyl)-6-ethoxy-2-methyl-, 4,5-bis(1,1-dimethylethyl)-6-ethoxy-6-oxido-2-phenyl-3,7-dioxa-4-aza-6-phosphanon-1-yl ester, 6-oxide (9CI) (CA INDEX NAME)

IC ICM C07C239-20

ICS C07F009-40; C07D251-32; C08F004-00

CC 35-3 (Chemistry of Synthetic High Polymers)

Section cross-reference(s): 29

300811-94-3P 300811-95-4P 310878-79-6P

310878-80-9P 310878-81-0P 310878-82-1P

5

310878-83-2P

(production of alkoxyamine phosphonates for use as radical polymerization catalysts)

REFERENCE COUNT:

THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE

IN THE RE FORMAT

L26 ANSWER 19 OF 19 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

2000:593981 HCAPLUS

DOCUMENT NUMBER:

133:297016

TITLE:

ľΤ

Macromolecular engineering using novel

alkoxyamines

AUTHOR(S):

Granou, Yves; Robin, Sophie; Guerrer, O.;

Couturier, J. L.

CORPORATE SOURCE:

Laboratoire de Chimie des Polymeres

Organiques, ENSCPB-CNRS-Universite Bordeaux 1

(UMR 5629), Talence, 33402, Fr.

SOURCE:

Polymer Preprints (American Chemical Society, Division of Polymer Chemistry) (2000), 41(2),

1352-1353

CODEN: ACPPAY; ISSN: 0032-3934

PUBLISHER:

American Chemical Society, Division of Polymer

Chemistry

DOCUMENT TYPE:

Journal

LANGUAGE:

English

AB Mono, di, and trifunctional alkoxyamines based on N-tert-butyl-N-(1-diethylphosphono-2,2-dimethyl)propyl nitroxide were used to polymerize styrene and Bu acrylate, affording well-defined of linear and star structures. Triblock as well as star block copolymers constituted of poly(Bu acrylate) inner part and polystyrene outer shell were subsequently synthesized by sequentially polymerization of these two monomers, using dialkoxyamine and trialkoxyamine as initiators.

IT 300811-94-3 300811-95-4

(catalysts; polymerization of styrene and Bu acrylate in presence of alkoxyamine catalysts)

RN 300811-94-3 HCAPLUS

CN 3,7-Dioxa-4-aza-6-phosphanonanoic acid, 4,5-bis(1,1-dimethylethyl)-6-ethoxy-2-methyl-, 1,6-hexanediyl ester, 6,6'-dioxide (9CI) (CA INDEX NAME)

RN 300811-95-4 HCAPLUS

CN 3,7-Dioxa-4-aza-6-phosphanonanoic acid, 4,5-bis(1,1-dimethylethyl)-6-ethoxy-2-methyl-, (2,4,6-trioxo-1,3,5-triazine-1,3,5(2H,4H,6H)-triyl)tri-2,1-ethanediyl ester, 6,6',6''-trioxide (9CI) (CA INDEX NAME)

PAGE 1-A

PAGE 1-B

CC 37-3 (Plastics Manufacture and Processing)

IT 300811-93-2 300811-94-3 300811-95-4

(catalysts; polymerization of styrene and Bu acrylate in presence of alkoxyamine catalysts)

REFERENCE COUNT:

20

THERE ARE 20 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

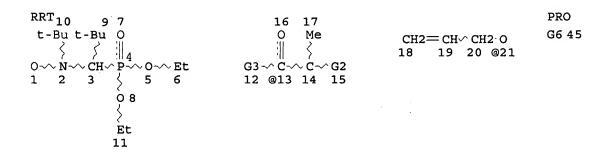
=> fil casreact

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L27

STR



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VAR G2=H/ME
VAR G3=21/25/28/31
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GRAPH ATTRIBUTES: RING(S) ARE ISOLATED OR EMBEDDED NUMBER OF NODES IS 45

STEREO ATTRIBUTES: NONE

L29 8 SEA FILE=CASREACT SSS FUL L27 ( 57 REACTIONS)

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L29 ANSWER 1 OF 8 CASREACT COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

143:347038 CASREACT

TITLE:

SG1 based alkoxyamines as radical initiators for the synthesis of lactones and lactams

Bertin, Denis: Gigmes, Didier: Margue, Sylvain

AUTHOR(S):

Bertin, Denis; Gigmes, Didier; Marque, Sylvain

R. A.; Tordo, Paul

CORPORATE SOURCE:

UMR 6517, Universites d'Aix-Marseille I, II et

III-CNRS, Marseille, 13397, Fr.

SOURCE:

Tetrahedron (2005), 61(36), 8752-8761

CODEN: TETRAB; ISSN: 0040-4020

PUBLISHER:

Elsevier B.V.

DOCUMENT TYPE:

Journal

LANGUAGE:

English

AB Recently, it has been shown that alkoxy amine derivs. can be used as radical initiators in tin-free radical chemical Thus, a method for the preparation of highly valuable alkoxyamines was developed, which involved ionic chemical and wherein a radical cyclization was triggered by thermal initiation. Following that procedure, bicyclic, spiro and eight-membered lactones were easily prepared in good yields with very high stereoselectivity. The reaction mechanism was discussed.

RX(1) OF 26 ... A + B ===> C...

C YIELD 40%

RX(1) RCT A 540770-92-1

STAGE(1)

SOL 75-09-2 CH2Cl2 CON 10 hours, reflux

STAGE (2)

RGT D 7719-09-7 SOC12

CON 45 minutes, room temperature

STAGE (3)

RCT B 107-18-6

RGT E 121-44-8 Et3N, F 1122-58-3 4-DMAP

USHA SHRESTHA EIC 1700 REM 4B28

# CON 16 hours, room temperature

PRO C 763105-79-9

REFERENCE COUNT:

48

THERE ARE 48 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L29 ANSWER 2 OF 8 CASREACT COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

143:26936 CASREACT

TITLE:

Long-range polar effect on the C-ON bond

homolysis in (tert-butyl[1-(diethylphosphonyl)-

2,2-dimethylpropyl]-aminoxyl) SG1-based

alkoxyamines

AUTHOR (S):

Bertin, Denis; Gigmes, Didier; Marque, Sylvain R. A.; Milardo, Stephan; Peri, Jerome; Tordo,

Paul

CORPORATE SOURCE:

Universite de Provence, UMR 6517, Marseille,

13397/20, Fr.

SOURCE:

Collection of Czechoslovak Chemical

Communications (2004), 69(12), 2223-2238

CODEN: CCCCAK; ISSN: 0010-0765

PUBLISHER:

Institute of Organic Chemistry and

Biochemistry, Academy of Sciences of the Czech

Republic

DOCUMENT TYPE:

Journal English

LANGUAGE:

Alkoxyamines and persistent nitroxyl radicals are important regulators of nitroxide-mediated radical polymerization. Because the polymerization times decrease with the increasing rate constant of the homolysis of the C-ON bond between the polymer chain and the nitroxyl moiety, the factors influencing the cleavage rate constant are of considerable interest. Because alkyl acrylate monomers are among the most used in polymerization, we present the measures of the rate consts. (kd) of the C-ON bond cleavage for new SG1 based alkoxyamine models containing para-substituted aromatic acrylates (4-XC6H4OC(O)C(Me)H-SG1). It appears that the values of kd increase with the electron-withdrawing properties of the para-substituent groups (4-X) of the ester SG1-based alkoxyamines.

RX(1) OF 7 A + B ===> C

В

(1)

A

C YIELD 43%

## RX(1) RCT A 540770-92-1

STAGE(1)

RGT D 7719-09-7 SOC12 SOL 75-09-2 CH2C12

CON 45 minutes, room temperature

STAGE (2)

RCT B 98-54-4

RGT E 121-44-8 Et3N CAT 1122-58-3 4-DMAP

72

SOL 60-29-7 Et20

CON 4 hours, room temperature

PRO C 852920-85-5

REFERENCE COUNT:

THERE ARE 72 CITED REFERENCES AVAILABLE

FOR THIS RECORD. ALL CITATIONS AVAILABLE

IN THE RE FORMAT

L29 ANSWER 3 OF 8 CASREACT COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

142:261127 CASREACT

TITLE:

Polar, Steric, and Stabilization Effects in

Alkoxyamines C-ON Bond Homolysis: A

Multiparameter Analysis

AUTHOR(S):

Bertin, Denis; Gigmes, Didier; Marque, Sylvain

R. A.; Tordo, Paul

CORPORATE SOURCE:

UMR 6517 Chimie Biologie et Radicaux Libres,

Universite de Provence, Marseille, 13397, Fr. Macromolecules (2005), 38(7), 2638-2650

SOURCE:

CODEN: MAMOBX; ISSN: 0024-9297

PUBLISHER:

American Chemical Society

DOCUMENT TYPE:

Journal

LANGUAGE:

English

We present measurements of the rate consts. (kd) of the C-ON bond cleavage in new alkoxyamine models containing the N-(2-methyl-2-propyl)-N-(1-diethylphosphono-2,2-dimethylpropyl)-N-oxyl (SG1) moiety. The homolysis rate consts. of 2,2,6,6-tetramethylpiperidinyl-1-oxyl (TEMPO)- and SG1-based alkoxyamines are analyzed in terms of polar inductive/field ( $\sigma$ U), steric ( $\sigma$ U), and radical stabilization ( $\sigma$ RS) contributions of the leaving alkyl radicals, using a multiparameter equation, i.e.,  $\log(kd/kd,0) = \rho U\sigma U + \delta \upsilon +$ 

ρRSσRS. The rate consts. increase with increasing

electron withdrawing, steric, and stabilization demands of the leaving alkyl radicals. Good correlations are found for TEMPO (log(kd/kd,0) = 13.6σU + 6.6υ + 13.9σRS) and SG1 (log(kd/kd,0) = 19.5σU + 7.0υ + 15.3σRS) derivs., highlighting the polar sensitivity of the leaving alkyl radical to the nitroxyl moiety. Such correlations should facilitate the design of new alkoxyamines as initiators/regulators and help to improve the tuning of NMP expts.

RX(1) OF 2 2 A + 2 B ===> C + D

RCT A 188526-94-5, B 80-58-0

RGT E 3030-47-5 PMDETA

PRO C 845738-91-2, D 845738-92-3

CAT 7440-50-8 Cu, 7787-70-4 CuBr

SOL 71-43-2 Benzene

CON 3 hours, room temperature

NTE 70% overall yield

REFERENCE COUNT:

115 THERE ARE 115 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L29 ANSWER 4 OF 8 CASREACT COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

141:260134 CASREACT

TITLE:

D

RX (1)

Stereochemical Studies of Chiral Acyclic Nitroxides Coupling with a Prochiral Radical

AUTHOR(S):

Braslau, Rebecca; Chaplinski, Vladimir;

Nilsen, Aaron; Arulsamy, Navamoney

CORPORATE SOURCE: Department of Chemistry and Biochemistry,

University of California, Santa Cruz, CA,

95064, USA

SOURCE: Synthetic Communications (2004), 34(13),

2433-2442

CODEN: SYNCAV; ISSN: 0039-7911

PUBLISHER: Marcel Dekker, Inc.

DOCUMENT TYPE: Journal LANGUAGE: English

AB Acyclic chiral nitroxides bearing a hydrogen atom on the carbon

adjacent to the nitroxide nitrogen were studied for

stereoselectivity in a coupling reaction with the prochiral

radical 1-phenethyl. In one case, an x-ray structure of the major

diastereomer was obtained, which corroborates a model for

predicting the stereoselectivity.

## RX(11) OF 16 A + AB ===> AC

AC YIELD 42%

RX(11) RCT A 585-71-7

STAGE (1)

RGT E 302-01-2 N2H4

CON 30 minutes, room temperature

STAGE (2)

RCT AB **188526-94-5** RGT F 1309-60-0 PbO2

SOL 108-88-3 PhMe

CON SUBSTAGE(1) 5 minutes, room temperature

SUBSTAGE(2) room temperature -> -78 deg C SUBSTAGE(3) -78 deg C -> room temperature

PRO AC 224575-62-6

NTE ultrasound, stereoselective COUNT: 14 THERE ARE 14

REFERENCE COUNT:

THERE ARE 14 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L29 ANSWER 5 OF 8 CASREACT COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 141:6742 CASREACT

TITLE: Diastereomeric excess upon cleavage and

reformation of diastereomeric alkoxyamines

AUTHOR(S): Ananchenko, Gennady; Marque, Sylvain; Gigmes,

Didier; Bertin, Denis; Tordo, Paul

CORPORATE SOURCE: Physikalisch-Chemisches Institut, Universitaet

Zuerich, Zurich, 8057, Switz.

SOURCE: Organic & Biomolecular Chemistry (2004), 2(5),

709-715

CODEN: OBCRAK; ISSN: 1477-0520

PUBLISHER: Royal Society of Chemistry

DOCUMENT TYPE: Journal LANGUAGE: English

The thermal reactions of several TEMPO and DEPN (N-(2-methylpropan-2-yl)-N-(1-diethylphosphono-2,2-dimethylpropyl)aminoxyl) based alkoxyamines were studied by means of 1H and 31P NMR spectroscopy aiming to distinguish the contributions of diastereoselective homolysis and coupling to the total equilibrium diastereomeric distribution. The TEMPO-based compds. reveal no diastereomeric excess while DEPN based compds. show a moderate excess both upon homolysis and coupling. The diastereomeric preference of homolysis for DEPN-(propionate-like) adducts does not depend on the ester group and it is mainly affected by the size of  $\beta$ -substituents. The diastereoselective coupling is sensitive to the total recombination rate constant kc and diastereoselectivity increases with the decrease of kc. Small diastereoselective coupling is found in the recombination of DEPN with sec-Bu isobutyrate radical, where no prochiral centers are formed upon cleavage of corresponding alkoxyamine.

RX(3) OF 8 ...K + L ===> M

YIELD 97%

RX(3) RCT K 188526-94-5

STAGE(1)

RGT F 3030-47-5 PMDETA, G 7787-70-4 CuBr, H 7440-50-8

Cu

SOL 67-64-1 Me2CO

CON room temperature

STAGE(2)

RCT L 736137-70-5

CON 18 hours, 50 - 55 deg C

PRO M 695230-19-4 NTE stereoselective

REFERENCE COUNT:

THERE ARE 53 CITED REFERENCES AVAILABLE 53

FOR THIS RECORD. ALL CITATIONS AVAILABLE

IN THE RE FORMAT

L29 ANSWER 6 OF 8 CASREACT COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

140:111235 CASREACT

TITLE:

Alkoxyamine-Mediated Radical Synthesis of

Indolinones and Indolines

AUTHOR (S):

Leroi, Corinne; Bertin, Denis; Dufils, Pierre-Emmanuel; Gigmes, Didier; Marque, Sylvain; Tordo, Paul; Couturier, Jean-Luc;

Guerret, Olivier; Ciufolini, Marco A.

CORPORATE SOURCE:

CNRS UMR 5622, Universite Claude Bernard Lyon 1 and Ecole Superieure de Chimie Physique Electronique de Lyon, Villeurbanne, 69622, Fr.

SOURCE:

Organic Letters (2003), 5(26), 4943-4945

CODEN: ORLEF7; ISSN: 1523-7060

PUBLISHER:

American Chemical Society

DOCUMENT TYPE:

Journal

LANGUAGE:

English

Thermolysis of  $\alpha$ -alkoxyamino propionanilides produces indolinones, whereas thermal reaction of N-allylaniline derivs. with various Tordo-type alkoxyamines results in formation of

indolines in the radical regime.

...A + B ===> C... RX(1) OF 67

C YIELD 90%

RCT A 2620-11-3, B 462104-38-7 RX (1)

D 7787-70-4 CuBr, E 5961-59-1 Benzenamine, RGT

4-methoxy-N-methyl-, F 7440-50-8 Cu

PRO C 647018-70-0 SOL 71-43-2 Benzene

CON 6 hours, room temperature

15

REFERENCE COUNT:

THERE ARE 15 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE

IN THE RE FORMAT

L29 ANSWER 7 OF 8 CASREACT COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

138:368316 CASREACT

TITLE:

AUTHOR(S):

Alkoxyamine-Mediated Radical Cyclizations Leroi, Corinne; Fenet, Bernard; Couturier,

Jean-Luc; Guerret, Olivier; Ciufolini, Marco

CORPORATE SOURCE:

Laboratoire de Synthese et Methodologie Organiques CNRS UMR 5078 and Laboratoire de Resonance Magnetique Nucleaire, Universite Claude Bernard Lyon 1 and Ecole Superieure de

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PUBLISHER:

American Chemical Society

DOCUMENT TYPE:

Journal

LANGUAGE:

English

GI

$$Me_3C$$
 $N-R$ 
 $H_2C$ 
 $X$ 
 $I$ 
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AB A new "conjunctive" radical cyclization process that involves the reaction of a 1,6-diene with the Tordo alkoxyamine, an agent originally developed for the radical polymerization of certain olefins through the "persistent radical effect", is developed. For example, thermolysis of non-conjugated dienes I [X = 0, CH2, (Eto2C)2C, PhCH2OCON, 4-BrC6H4SO2N] in the presence of Tordo alkoxyamine gave cycloadducts II [R = (EtO)2POCH(CMe3)] predominantly as cis-isomers in 25-51% yields. Acidic cleavage of II [R = (EtO)2POCH(CMe3)] afforded the corresponding hydroxylamines II (R = H) in good yields (74-99%).

RX(1) OF 18 A + B ===> C...

YIELD 25%

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RX(1)
         RCT A 462104-38-7, B 3070-53-9
         PRO C 521276-79-9
         SOL 75-65-0 t-BuOH
         CON 60 hours, 120 deg C
         NTE stereoselective
                        36
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REFERENCE COUNT:

THERE ARE 36 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L29 ANSWER 8 OF 8 CASREACT COPYRIGHT 2006 ACS on STN ACCESSION NUMBER: 133:309685 CASREACT TITLE: Method for preparing alkoxyamines from nitroxides

INVENTOR(S): Couturier, Jean-Luc; Guerret, Olivier;

Senninger, Thierry

PATENT ASSIGNEE(S): Elf Atochem S. A., Fr. SOURCE: PCT Int. Appl., 25 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent LANGUAGE: French

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

PA	PATENT NO. KIND DATE														
WO	2000										-			2000	0324
	W:	ΑE,	AL,	AM,	ΑT,	AU,	ΑZ,	BA,	BB,	BG,	BR,	BY,	CA,	CH,	CN,
		CR,	CU,	CZ,	DE,	DK,	DM,	EE,	ES,	FI,	GB,	GD,	GE,	GH,	GM,
		HR,	ΗU,	ID,	IL,	IN,	IS,	JP,	KΕ,	KG,	ΚP,	KR,	ΚZ,	LC,	LK,
		LR,	LS,	LT,	·LU,	LV,	MA,	MD,	MG,	MK,	MN,	MW,	MX,	NO,	NZ,
		PL,	PT,	RO,	RU,	SD,	SE,	SG,	SI,	SK,	SL,	ТJ,	TM,	TR,	TT,
		TZ,	UA,	UG,	US,	UΖ,	VN,	ΥU,	ZA,	ZW,	AM,	ΑZ,	BY,	KG,	KZ,
		MD,	RU,	ТJ,	$\mathbf{T}\mathbf{M}$										
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		CY,	DE,	DK,	ES,	FI,	FR,	GB,	GR,	ΙE,	IT,	LU,	MC,	NL,	PT,
		SE,	BF,	ВJ,	CF,	CG,	CI,	CM,	GA,	GN,	GW,	ML,	MR,	NE,	SN,
		TD,	TG	•											•
FR	2791	979		A:	1	2000	1013		F	R 19	99-4	105		1999	0408
	2791														
CA	2334	845		A.	Ą	2000	1019		CZ	A 20	00-2	33484	45	2000	0324
BR	2000	0060													
EP	1086	073		A:	L	2001	0328		E	200	00-9	1425	1	2000	0324
EP	1086	073		В:	1	2003	0312								
	R:	AT,	BE,	CH,	DE,	DK,	ES,	FR,	GB,	GR,	IT,	LI,	LU,	NL,	SE,
		MC,	PT,	ΙE,	FΙ										
JP	2002	5412	39	T	2	2002	1203		JI	200	00-6	1082	1	2000	0324
	2342									Γ 20	00-91	1425	1 ′	2000	0324
ES	2193	946		T:	3	2003	1116		ES	3 20	00-9	1425	1.	20000	0324
	2000														
BG	1050	35		Α		2001	0731		В	3 20	00-1	05035	5	2000	1208
US	6495	720		В:	l	2002	1217		US	3 200	01-7	19042	2	2001	0223
PRIORIT	Y APP	LN.	INFO	<b>. :</b>					FI	R 19	99-44	105		19990	0408
								•	W	200	00-F	3750		20000	0324

OTHER SOURCE(S): MARPAT 133:309685

The invention concerns a method for preparing alkoxyamines which consists in mixing in an organic solvent, a metallic salt, a metal ligand, a halo-carbon ZX compound, and a nitroxide; in maintaining the reaction medium stirred at a temperature ranging between 20 >C and 90 >C, until the nitroxide disappears; in recuperating the organic by evaporating the organic solvent under reduced pressure. E.g., reaction of TEMPO with PhCHBrMe in presence of Cu, CuBr and N,N,N',N',N'',-pentamethyldiethylenetriamine gave 97% 1-(2,2,6,6-tetramethylpiperidinyloxy)-1-phenylethane.

RX(1) OF 3 A + B ===> C

Α

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 & EtO-P-CH-Bu-t \\
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C YIELD 99%

RX(1) RCT A 585-71-7, B 188526-94-5

STAGE (1)

CAT 3030-47-5 PMDETA, 7787-70-4 CuBr SOL 108-88-3 PhMe

STAGE(2)

RGT D 7732-18-5 Water

PRO C 288583-77-7

NTE USING ANOTHER LIGAND GAVE TWO ISOMERS

REFERENCE COUNT:

THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT